STUDIES ON THE PHYSICAL PROPERTIES AND REDUCTION-SWELLING BEHAVIOR OF FIRED HAEMATITE IRON ORE PELLETS

**A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENT FOR THE DEGREE OF**

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**In
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**By
Rajnish Kumar
(Roll No: 211MM1197)**

**Department of Metallurgical and Materials Engineering
National Institute Of Technology, Rourkela**
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Date: **Rajnish Kumar**

Place: **(Roll No:211MM1197)**

<u>CERTIFICATE</u>

This is to certify that the project report entitled **"STUDIES ON THE PHYSICAL PROPERTIES AND REDUCTION-SWELLING BEHAVIOR OF FIRED HAEMATITE IRON ORE PELLETS"** submitted to **"National Institute of Technology, Rourkela"** in partial fulfilment of the requirement for the award of the Degree of **"Master of Technology"** is an authentic and original work carried out by "***Rajnish Kumar***" with Enrolment No**. "*211MM1197*"** under the guidance of ***Prof. M Kumar***

The matter embodied in this project is genuine work done by the student and has not been submitted whether to this University or to any other University / Institute for the fulfilment of the requirement of any course of study.

(Signature of the Student)

(Signature of the Guide)
Prof. M. Kumar
Dept of Metallurgy & Material Engg
NIT, Rourkela

ABSTRACT

In view of generation of huge amount of iron ore and coal fines there is a need to develop a technique to utilize these fines. The present project work has been undertaken to study reduction and swelling characteristics of hematite iron ore pellets made from iron ore fines under different conditions. The physical properties of fired iron ore pellets were studied. The compressive strength and drop number of fired pellets increased with the increase of firing temperature due to the enhancement in the extent of sintering and consolidation of iron ore fines.

The main objectives of the present work have been to study the effects of (i) Reduction temperature and time (ii) Addition of concentrated sugarcane juice on the reduction and swelling behaviours of fired iron ore pellets. The results indicated a decrease in the extent of reduction with increase of firing temperature. The reduction rate of fired iron pellets were found to increase with rise of reduction temperature from (800°C-950°C). The degree of reduction of fired pellet increased with increase of reduction time, the rate being faster in the initial 20-30 minutes followed by a decrease thereafter. Reduction at a temperature of 850°C, in general, gave lowest degree of swelling in the reduced pellets. The highest degree of swelling at a reduction temperature of 850°C appears to be due to fibrous growth of iron in the reduced pellets. Slightly lower values swelling in the fired iron ore pellets reduced at 900°C and 950°C appear to be due to sintering of iron particles in the pellets, as indicated by their SEM micrographs. Effect of reduction time in the range studied on the extent of swelling was not found to be distinguishable.

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INTRODUCTION

1. INTRODUCTION

The sponge iron has of late come up as a major input material for steel making through electric furnace route-both electric arc furnace (EAF) and electric induction furnace (EIF). Because of the long gestation period, large investments, dependence on coke and moreover on the foreign suppliers etc. The steel industry is slowly inclining towards the electric arc furnace route from blast furnace route and that is why the requirement of sponge iron is increasing very fast. Sponge iron is a better substitute for scrap for steelmaking through EAF/EIF routes due to its homogenous (same chemical composition) nature, improved productivity and lower coke consumption. Majority of fines which are generated during the course of mining, handling, transportation etc. are also exported at a through away price (discarded after use), which need to be utilized by making pellets of iron ore for making the sponge iron. According to industry experts, the preference for usage of direct reduced iron (DRI) will lead to the use of 80 percent DRI in the charge mix in EAFs by 2009-10 which may even reach the 85 percent level by 2011-12 in the various regions of India [1].

1.1 DETAILS OF DRI PRODUCTION IN INDIA AND IN THE WORLD

A year-wise production of sponge iron in the world including India is given in Table-1.1. According to current analysis, it has been observed that starting with a meager production of 0.79 million tons in 1970, the world sponge iron production went up to 55.85 million tons in 2005, as given in Table1.it is clear from the table that the world sponge iron production has been increased nearly 225% from the year 1990 to 2005. In the year 2002 India became the largest producer of sponge iron in the world with a production of 5.48 million tones and still it has retained its first slot in the world rating of sponge iron production. Out of 16.27 MT of sponge iron produced in 2006-07 in that amount the contribution of coal based sponge iron units is around 11.01 MT and of based units are 5.26 MT. This large difference in contribution of G.B.S.I.U. and C.B.S.I.U. is due to shortage (acute availability) of natural gas and profuse availability of non-coking coal in India. This phenomenal growth of DRI industries is driven by increasing demand of steel in India and as well as in the world. Now India has ranked a sixth largest steel producer in the world with a production of 42 MT/Annum.

TABLES

Table-1.1

Year wise Sponge Iron Production in World and in India

Year	World scenario		Indian scenario	
	Production (MT)	Growth (%)	Production (MT)	Growth (%)
1990-91	17.68	_	NA	_
1991-92	19.32	9.27	1.31	NA
1992-93	20.51	6.15	1.44	9.92
1993-94	23.65	15.30	2.40	66.66
1994-95	27.37	15.70	3.39	41.25
1995-96	30.67	12.00	4.40	29.79
1996-97	33.30	8.40	5.00	13.63
1997-98	36.19	8.88	5.30	6.00
1998-99	36.96	2.50	5.22	-1.50
1999-00	38.60	4.10	5.34	22.98
2000-01	43.78	11.90	5.48	26.21
2001-02	40.32	-6.99	5.43	-9.12
2002-03	45.08	12.00	6.9	27.07
2003-04	49.45	9.69	8.08	17.10
2004-05	54.60	10.41	10.30	27.45
2005-06	55.85	2.23	11.47	11.35
2006-07	59.8	_	16.27	_
2007-08	68.5	_	20	_

Source: steelworld.com

1.2 IRON ORE AND COAL RESERVES IN INDIA

Presently, India's number is fifth in terms of iron ore reserves. It has got 25 billion tons of reserves, of Which 15 billion tons are reported to be hematite and rest magnetite at cut off grades of 55% iron as per Indian bureau of mines (IBM). India produces around 155 million tons of iron ore, including both lumps and fines, in which about 52 million tons were used by the domestic steel manufacturers.

Table 1.21 Iron Ore Reserves in India

States	Main Ore	Fe Range (%age)	Alumina (%age)	Phos Max (%age)
A-Orissa, Jharkhand	Haematite	62-64	2-4	0.04-0.1
B-Chattisgarh, MP, Maharashtra	Haematite	64-66	1.0-4.0	0.04-0.15
C-Karnataka	Haematite	62-64	2.0-4.0	0.04-0.09
D-Goa	Haematite	60-63	2.0-4.0	0.04-0.07
E-Karnataka	Magnetite	35-45	1.0	-

States	Major Mines / Deposits
A-Orissa, Jharkhand	Chiria, Noamundi, Joda, Kiriburu, Meghataburu, Thakurani, Bolani, Gua, Malangtoli, Gandhamardan, Daitari
B-Chattisgarh, MP, Maharashtra	Bailadila, Dalli,Rajhara, Rowghat, Mahamaya, Aridongri, Surajgarh
C-Karnataka	Donimalai, Ramandurg, Kumaraswamy, NEB Range, Ettinahatti, Tumti, Belagal
D-Goa	N Goa, S Goa, Redi
E-Karnataka	Kudremukh, Bababudan, Kudachadri

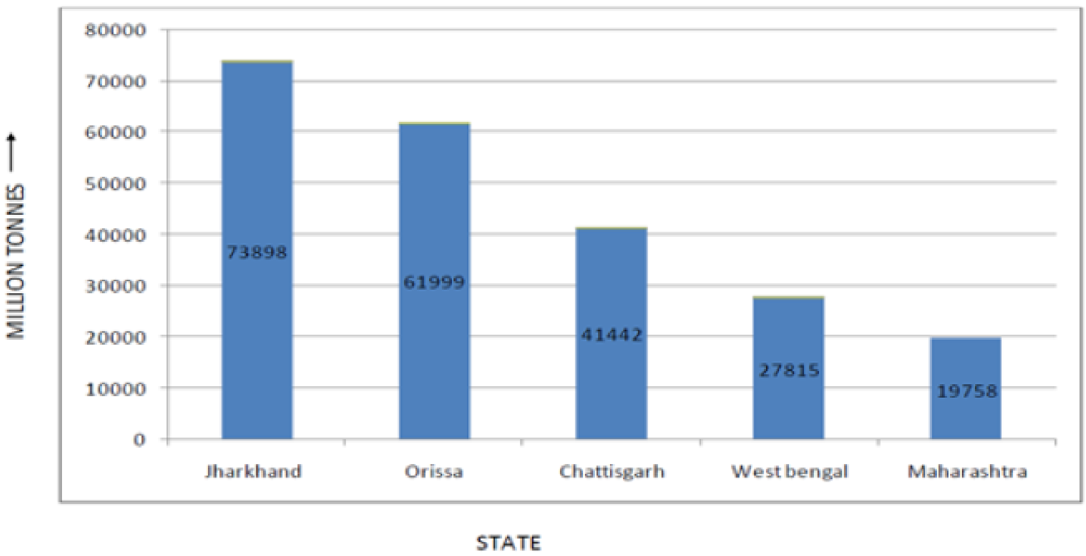
Table 1.22

		2000-01	2001-02	2002-03	2003-04	2004-05	2005-06(P)
Crude steel production	(A) Main producers	17254	17762 (2.94)	18982 (6.87)	20012 (5.43)	20015 (0.01)	21694 (8.39)
	(B) Secondary producers	9703	10202 (5.14)	11461 (12.34)	14236 (24.21)	14806 (4.00)	19650 (6.38)
	TOTAL	26957	27964 (3.73)	30443 (8.86)	34248 (12.50)	34821 (1.67)	41344 (7.43)
Iron ore consumption		36020	37713 (4.70)	40936 (8.54)	44974 (9.86)	48150 (7.06)	52523 (9.08)
Iron ore exports		37270	41640 (11.72)	48020 (15.32)	62570 (30.30)	78145 (24.89)	89277 (14.25)
Iron ore production		80762	86226 (6.76)	99072 (14.90)	122838 (23.99)	145942 (18.81)	154436 (5.82)
Surplus iron ore		7472	6873	10116	15294	19647	12636

Source : 1 Joint Plant Committee, Kolkata
2 Indian Bureau of Mines, Nagpur

India's number is third in the largest producers of coal and holds the fourth position in the largest coal reserves in the world consisting 10% of the world share of coal reserves. Percentage contribution of non coking coal to about 85% i.e. 221 billion tons, while rest 15% i.e. 32 billion tons amount is having by coking coal. Indian coal has high ash content (15-45%) and low calorific value.

Fig 1.3 Coal state wise distribution



The sponge iron industry is quite comfortable so far the reserves of iron ore and coal are concerned. Amount of total recoverable resources of iron ore in the country account for about

9% of the world's reserves and the major deposits are located in the states of Orissa (34%), Jharkhand (27%), Chhattisgarh (18%), etc. Grade-wise total proved reserves of non-coking coal in India have been outlined in Table 1.31 below. The gradation of Indian non-coking coals has been carried out on the basis of useful heat value (UHV). In Table1.32 [2] the reserves and their distribution is shown. As per the present system of coal linkage in India, about 80% of the production of high grade coals (A, B and C) goes to the power sector and the balance 20% to other industrial sectors including sponge iron plants .These data clearly indicate that measures must be taken to utilize the lower grades of coal like D, E, F and G through the better process control.

1.3MAJOR ROUTES OF STEEL MAKING

Primarily there are two routes of steel making;

1)BF-BOF 2)DR-EAF

Nowadays DR-EAF route is overwhelming the BF-BOF route and contributes around 35% of the total World's steel production at present.

1.3.1Shortcomings of the BF-BOF Route

- Limited availability of coking coal reserves in India.
- Longer gestation period
- Large investment cost and heavy emissions of pollutants
- Import cost of coking coal is too much
- Its poor flexibility in production capacity.

1.3.2Advantagesof DR-EAF Route over BF-BOF

- In the plant operation, its simplicity leads the way over BF-BOF
- Flexibility to operate at smaller capacities, down to 300,000 ton/year with an attractive production cost.

- Eco friendly impact compared to coking plants and blast furnaces
- Then realization of new plants can be manage in phase and in turn financial structure can be optimized.
- This route can operate with natural gas, non cocking coal, petrochemical wastes etc. and that is why possibility of using local energy sources enhances.
- The most dominating parameter of DR-EAF over BF-BOF [3] is its very attractive investment cost.

1.4 Thermodynamics and Kinetics of Iron Ore Reduction

Chemical Reactions and Thermodynamic Analysis

Coal contains volatile matter and the iron oxide i.e. Fe_2O_3 [4] On heating, the following reactions take place;

Coal → char + tar + gases (CO, CO_2, H_2, C_nH_m and N_2) …... …………….(1)

$Fe_2O_3(s) + 3C(s) \rightarrow 2Fe(s) + 3CO(g)$ …………………………. (2)

Reduction of hematite : $Fe_2O_3(s) + CO(g) / H_2(g) \rightarrow 2Fe_3O_4(s) + CO_2(g) / H_2O(g)$ … ..(3)

Reduction of magnetite : $Fe_3O_4(s) + CO(g) / H_2(g) \rightarrow 3FeO(s) + CO_2(g) / H_2O(g)$ …….(4)

Reduction of wustite: $FeO(s) + CO(g) / H_2(g) \rightarrow Fe(s) + CO_2(g) / H_2O(g)$ ………… ….. (5)

Besides the above mentioned reactions, the next three possible reactions are :

$C(s) + CO_2(g) \rightarrow 2CO(g)$ …………. (6)

$C(s) + H_2O(g) \rightarrow H_2(g) + CO(g)$.. …(7)

Reaction (1) represents thermal decomposition (devolatilization) of coal and it starts in the temperature range of 300 – 500^0C. The products (CO & H_2) of reactions (1) and (2) induce reactions (3), (4) and (5). Carbon gasification reactions with CO_2 and H_2O (6& 7) occur at temperatures above 800^0C and generates reducing gases (CO & H_2) for the reduction of iron oxides . The gas, in excess to that required for the reactions, flows out from the reactor. On the basis of thermodynamic analysis from G vs. T and H vs. T plots for the reduction with CO and H_2 done by Komatina & Gudenau 2004, it could be said that the most probable reaction is (3), followed by reactions (4) and (5). The reactions (6) and (7) are highly endothermic and possible at higher temperatures. In comparison to CO gas, the reduction reactions with H_2 gas are mostly endothermic and favoured at higher temperatures.

1.5Kinetic Steps Involved during Iron Ore Reduction

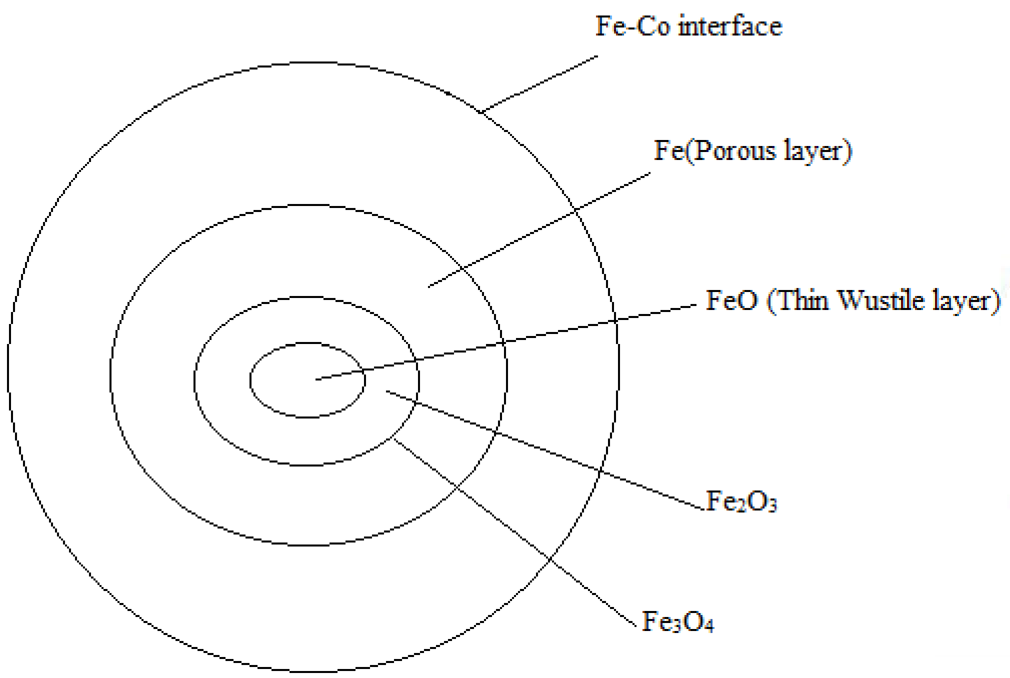


Fig.1: Schematic diagram in modes of reduction of iron oxide

Steps:-

1.Transport of CO gas from bulk gas phase to Fe-CO interface.

2.Adsorption of CO gas at the Fe-CO interface.

3.Transport of CO gas from Fe-CO interface to Fe-Fe_2O_3 interface.

4.Adsorption of CO gas at the Fe-Fe_2O_3 interface.

5.Chemical reaction betweenFe_2O_3 and CO gas at the Fe- Fe_2O_3interface.

6.Fe_2O_3 + CO= Fe + CO_2

7.Desorption of the product gasCO_2 from Fe- Fe_2O_3interface to Fe-CO interface.

8.Transport of $CO_2$from Fe- Fe2O3 interface to Fe-CO interface.

9.Diffusion ofCO_2 from Fe-CO interface to the bulk gas phase.

10.Diffusion of tiny iron nuclei and merging into bigger nuclei.

The steps involved are either diffusion or chemical and the slowest of these control the overall rate of reaction.

1.6 Factors Affecting Reduction Kinetics of Iron Ore

The following factors affect the reduction kinetic of iron ore;

- Physical characteristics of the oxide ore (size, shape, porosity, etc.)
- Temperature of the charge
- CO/H_2 content in the gas phase
- Gas flow rate
- Chemical nature of the oxide
- Gangue content in the ore
- Reactivity of the solid fuel
- Pressure applied
- The extent of solid-solid/gas-solid reaction.

1.7. Objectives of the Project:

The following are the objectives of the present project work:

- Characterization of the physical and chemical properties of selected iron ore.
- Characterization of the properties of selected non-coking coals.
- Analysis of the effect of binder addition on the crushing strength, porosity and reduction characteristics of fired hematite iron ore pellets.
- Analysis of the effect of reduction temperature and time on the degree of reduction of fired hematite iron ore pellets.
- Study of the effect of time on extent of swelling of iron ore pellets.
- Study of effect of temperature on extent of swelling of iron ore pellets.
- Study of Correlation between Degree of reduction and Percentage swelling of iron ore pellets.
- X-Ray Diffraction (XRD) analysis of reduced iron ore pellets.
- Micro structural study (SEM) of reduced iron ore pellets.

CHAPTER-2

LITERATURE REVIEW

Literature review:

(1) Binding effects in hematite and magnetite concentrates: S. Komar Kawatra, Joseph A. Halt (2011) [5]

Kawatra& Halt pelletized both the hematite and magnetite with a binder, bentonite. . As-received magnetite and as-received hematite were pelletized and tested for wet-drop number and dry-crush strength. Hematite pellets exceeded industrial minimum wet-drop and dry-crush values of 5 drops and 22 N/pellet without bentonite addition, while magnetite pellets exceeded industrial minimum values at a bentonite dose of 6.6 kg/t (0.66%). It is known that finer particles increase pellet strength, So additional magnetite was ground to a similar particle size distribution as the as-received hematite. The ground magnetite was pelletized and tested for wet-drop number and dry-crush strength. Wet drop and dry crush values increased after grinding the magnetite concentrate. However, they were significantly less than hematite pellets at similar bentonite doses. Consequently, Particle size effects were not the dominant cause for higher strengths in the hematite concentrate.

(2)Characteristics of Indian non coking coals and iron ore reduction by their chars for directly reduced iron production: M. Kumar ; S. K. Patel (2008) [6]

Kumar & Patel found in their research work that Studies on chemical and physical properties (proximate analysis, sulphur content, reactivity, iron ore reduction potential, caking index and ash fusion temperatures) of coals, procured from sixteen different mines of Orissa, were undertaken for their judicial selection in Indian sponge iron plants. These coals were found to have low sulphur (range: 0.40 – 0.66 %) and moderate to high ash (range : 22 – 53 %) contents. The results indicated no caking characteristic in all the coals except Basundhara. Majority of the studied coal ashes were found to have higher fusion temperatures (ST: 1349 – 1547^0C;HT: 1500 – 1663^0C; and FT: 1510 – 1701^0C). Also an increase in fixed carbon content in the coal char, in general, led to decrease in its reactivity towards CO_2, and majority of the chars exhibited significantly higher reactivities (> 4.0 cc of CO/g.sec). Further reduction studies in coal chars at 9000C indicated an increase in the degree of reduction of fired hematite iron ore pellets with increase of char reactivity and reduction time. The authors recommend the utilization of majority of the studied coals as such and some of them(Lakhanpur, Samleshwari, Orient OC– 4 and Dhera coals) after blending or beneficiation.

(3) International Journal of Mineral Processing Volume 59, Issue 4: B.K Pandey and, T Sharma (July 2000) [7]

An attempt has been made by Pandeyand Sharma to study the effect of reducing agents (coke, Non-coking coal, Char, Charcoal, etc.) on the reduction behaviour of double-layered pellets consisting of a core of iron ore and reducing agent mixture within a shell of iron ore. The reduction tests were conducted under isothermal condition in the temperature range 1000–1200°C. The variables (parameters) studied were the reduction temperature, carbon/iron oxide ratio of the core, and reduction time. The experiments were statistically designed such that the effect of each variable and interactional effect of each variable can be quantitatively assessed and compared. The results show highest degree of reduction with non-coking coal followed by charcoal, char, coke-fines. Among these three parameters, reduction time has the strongest effect when charcoal and non-coking coals are used as reducing agents, whereas in the case of coke and char, reduction temperature has the strongest effect. C: Fe_2O_3 ratio of core has the least effect in all four types of carbonaceous core double-layered pellets.

(4) Pelletization of magnetite ore with colemanite added organic binders: O. Sivrikaya(2010) [8]

Sivrikaya has investigated using a new generation binder consisting of an organic binder and a borate salt was tested as an alternative to bentonite in magnetite ore pelletization. Carboxyl methyl cellulose (CMC), Ciba DPEP06-0007 and corn starch, calcined colemanitewere used as organic binders and the borate salt, respectively. Different combinations at several addition levels were added to pellet feed separately. Organic binders use is found to be sufficient in terms of wet pellet quality; however, they fail to render the required compressive strength to pre-heated and fired pellets. Therefore, organic binders and calcined colemanite were used together so that wet pellets, pre-heated and fired pellets would be of the required quality, The results showed that the use of an organic together with calcined colemanite indeed yielded pellets with equal or better wet and indurate pellet qualities compared to the pellets produced with bentonite binder alone.

(5) Reduction of double layered iron ore pellets:T. Sharma (1996) [4]

Sharma had made an attempt to study the reduction behaviour of double layer pellets consisting of a core of iron ore and a non- coking coal mixture within a shell of iron-ore.

Here tests were conducted under isothermal conditions in the temperature range 1000-1200°C. Reduction temperature, reduction time and carbon/iron oxide ratio are the parameters which have been studied. The experiments were statistically designed such that the effect of each variable and interactional effect of different variables can be quantitatively assessed. The effect of reduction temperature on degree of reduction is strongest are the results which is followed by reduction time and carbon/iron oxide ratio.

(6)Thermal investigations of direct iron ore reduction with coal: Gui-su Liu, Vladimir Strezov, John A. Lucas, Louis J. Wibberley(2003) [9]

Liu et al. have investigated using several advanced experimental techniques on the fundamental mechanisms for iron ore reduction in coal-mixtures. Initially the thermal properties of coal-mixtures were examined and apparent specific heat of coal-ore mixtures against temperature was obtained at heating rate 10° C/min. Several exothermic and endothermic peaks were observed which were related to the decomposition reactions and reduction. The flue gases from the mixture were analysed using a mass spectrometer. Secondly, the X-ray diffraction (XRD) and the iron phase analytical techniques were applied to identify the iron phase changes with the temperature. It has been found that coal devolatilisation and iron oxides reduction occur simultaneously during the heating of the mixture. H_2 and CO gases produced from coal devolatisation and char gasification were responsible for the reduction of iron oxides at these temperatures. Iron oxides undergo step-wise reduction over the whole process.

(7) Binding mechanisms in wet iron ore green pellets with a bentonite binder: S.P.E. Forsmo, A.J. Apelqvist, B.M.T. Björkman, P.O. Samskog. (2005) [10]

S.P.E.\ et.al investigated the process of sintering mechanism in magnetite iron ore pellets. From their experiments they concluded that Oxidation mechanisms and thermal volume changes in magnetite iron ore pellets as a function of raw material fineness and pellet porosity . When a pellet starts to oxidize, a shell of hematite is formed around the pellet while the core still is magnetite. Dilatation curves were measured under non-oxidizing and oxidizing atmospheres to separately describe thermal volume changes in these two phases. Dilatation measurements showed contraction during oxidation between 3300 and 900 °C by

0.52%. The extent of contraction was not influenced by raw material fineness or the original porosity in the pellets. Simultaneously with the contraction in the hematite shell, linear expansion in the magnetite core took place. Sintering started earlier in the magnetite core (950°C) compared to the hematite shell (1100 °C). The difference in sintering rates increased with increasing fineness in the magnetite concentrate. A finer grind in the raw material would, therefore, promote the formation of duplex structures.

(8) Characterization of properties and reduction behaviour of iron ores for application in sponge iron making: M. Kumar, S. Jena and S. K. Patel (2008)[2]

In this work**,** chemical and physical properties, and reduction behaviour (in coal) of hematite iron ores, procured from ten different mines of Orissa, were studied to provide information to the iron and steel industries (sponge iron plants in particular). Majority of the iron ores were found to have high iron and low alumina and silica contents. And also all these iron ores were free from the deleterious elements (S, P, As, Pb, alkalies, etc.). The results indicated lower values of shatter and abrasion indices, and higher values of tumbler index in all the iron ore lumps except Sakaruddin (previous) and Khanda Bandha OMC Ltd. For all the fired iron ore pellets, the degree of reduction in coal was more intense in the first 30 minutes after which it became small. Slow heating led to higher degree of reduction in fired pellets than rapid heating. All the iron ores exhibited more than 90% reduction in their fired pellets in 2 hrs time interval at a temperature of 900^0C. Iron ore lumps showed lower degree of reduction than the corresponding fired pellets.

(9) The role of normal and activated bentonite on the pelletization of barite iron ore concentrate and the quality of pellets: O.A. Mohamed, M.E.H. Shalabi, N.A. El-Hussiny, M.H. Khedr, F. Mostafa (2002) [11]

In this study, the essential parameters affecting the pelletization process of high barite iron ore concentrate were studied using the Egyptian normal and activated bentonite as binder materials. The metallurgical properties of green, dried and fired pellets were studied using chemical and X-ray analyses. The average strength of fired pellets containing 1.5% normal bentonite and fired at 1300^0C for 25 min exceeded 200 kg/pellet. Use of activated bentonite produced a lower strength in the pellet. Meanwhile, the productivity of green pellets decreased when the last binder was used.

(10)Assessment of reduction behaviour of hematite iron ore pellets in coal fines for application in sponge iron making: M. Kumar & S. K. Patel (2009) [12]

In this research work, isothermal reduction kinetics (with *F* grade coal) in fired pellets of hematite iron ores, procured from four different mines of Orissa, were carried out in the temperature range of 850–1000◦C to provide information for the Indian sponge iron plants. The rate of reduction in all the fired iron ore pellets increased markedly with a rise of temperature up to 950◦C, and thereafter it decreased at 1000◦C. The rate was more intense in the first 30 minutes. All iron ores exhibited almost complete reduction in their pellets at temperatures of 900 and 950◦C in <2 hours' heating time duration, and the final product morphologies consisted of prominent cracks. The kinetic model equation 1 − (1 − _)1/3 = *kt* was found to fit best to the experimental data, and the values of apparent activation energy were evaluated. Reductions of D. R. Pattnaik and M. G. Mohanty iron ore pellets were characterized by higher activation energies (183 and 150 kJ mol−1), indicating carbon gasification reaction to be the rate-controlling step. The results established lower values of activation energy (83 and 84 kJ mol−1) for the reduction of G. M. OMC Ltd. and Sakaruddin iron ore pellets, proposing their overall rates to be controlled by indirect reduction reactions.

CHAPTER-3

EXPERIMENTAL

3.EXPERIMENTAL

3.1 Selection of Material

The iron ore was procured from sakaruddin iron ore mine and non coking coal was collected from Basundhara mine of Odisha .The Sugar cane juice was collected from the local market.

3.2 Determination of Chemical Composition and Loss on Ignition of Iron Ore

The chemical composition of the iron ore was determined by X-ray fluorescence technique at Rourkela Steel Plant. The loss on ignition values of the iron ore was determined by heating 1gm. of air dried samples at a temperature of 900^0C for 1hr, followed by air cooling. Loss in weight was taken as the % loss in ignition.

3.3 Proximate Analysis of Non-Coking Coal

Analysis for moisture, volatile matter, ash and fixed carbon contents were carried out on samples ground to pass through 72 mesh B.S. test sieve as follows [2]:

3.3.1 Moisture Determination

1 gm. of air dried sample of – 72 mesh size was placed in an air oven maintained at a temperature of 110^0C and kept there for 1 hour. The loss in weight expressed as the percentage of initial weight of coal gives the percentage of moisture content in the sample.

3.3.2 Volatile Matter Determination

1 gm. of air dried sample – 72 mesh size was taken in a volatile matter crucible (made of silica) covered with a lid**.** The crucible was introduced in the furnace maintained at a temperature of 925^0C and kept at this temperature for 7 minutes. The crucible was then taken out and loss in weight of sample was determined. The % loss in weight minus % of moisture content in the sample gives the value of percentage volatile matter in the sample on air dried basis.

3.3.3 Ash Determination

1 gm. of air dried sample was taken in a Silica disc and placed in the furnace maintained at a temperature of 775^0C, and kept there till complete burning. The weight of ash obtained expressed as the % of initial weight of the sample gives % of content in the sample on air dried basis.

3.3.4 Fixed Carbon Determination

It was simply calculated as follows:

% Fixed Carbon = 100 - % (moisture + volatile matter + ash)

3.4 PREPARATION OF IRON ORE PELLET.

Binder

The sugarcane juice was heated at about 150°C for 4 hours inside oven to concentrate it to get better binding properties [13].

Pellet Preparation

Preparation of pellets of 15-17 mm size were made by hand rolling of a mixture of iron ore fines of -100 mesh size and -16+25 mesh size (moistened) in the ratios of 9.5:0.5 and 9:1 Pellets from Sakaruddin iron ore were made with the addition of binder, which is concentrated sugarcane juice (2, 4 and 6 wt % of iron ore). Then the pellets were kept inside the oven, which is maintained at 110°C for drying and removal of moisture for 4hr. After 4hr the pellets were taken out and kept in separate plastic pouches.

3.5 Firing of Pellets

The iron ore pellets were fired by heating them from room temperature to the predetermined firing temperatures (1100 °C) at a rate of about 7°C /minute. The soak time at this firing temperature was 1 hour. After soaking for 1 hour, the furnace was switched off and the pellets were allowed to cool in the furnace themselves. The pellets were taken out from the furnace and kept separately in different plastic pouches marked with pellet preparation conditions.

3.6 Evaluation of Physical Properties of Fired Iron Ore pellets

The produced fired iron ore pellets were processed for the determination of properties such as porosity and crushing strength values [14].

3.7 Determination of Apparent Porosity

The values of apparent porosity for fired iron ore pellets were determined by using kerosene oil as a medium in according to the given formulae;

Apparent porosity = W – D/ W- (S-s)

Where, ‘D’ is the weight of dried pellet;

‘W’ is the weight of oil saturated pellet;

‘S’ is the weight of the pellet + thread while immerse in oil;

And’ s’ is the weight of thread only while immerse in oil.

3.8 Determination of Cold Crushing Strength

By using a uni-axial hydraulic power press, crushing strength of the fired pellets and some of the reduced pellets have been determined. The size of reduced pellets is 15 mm.

Using following formulae, values of crushing strength were found and reported in results and discussion section of the same thesis work.

$\sigma_c = W/A$

Where, σ_c is the crushing strength in kg.cm^2;

W is the maximum load at fracture in kg;

And A is the area in cm^2

3.9 Procedure for Reduction Studies

The samples were heated in a muffle furnace from room temperature to the required reduction temperatures of 800, 850, 900, 950^{0}C heated at a rate of about 10^{0}C per minute and at these temperatures for varying time periods of 15, 30, 45, 60 & 90 minutes for the reduction temperatures of 800, 850 & 900^{0}C and 5, 10, 15 20 & 25 minutes for the reduction temperatures of 950^{0}C [15].

Weighed amount of air dried pellet of size 15mm was placed on a packed bed of non-coking coal crushed into a size of 2-3mm in a stainless steel container (size: 75mm height × 39mm inside diameter) with a mouth closed tightly by an air tight cover having an outlet for exit of gases. The iron ore pellet was positioned in the middle, in the packed bed of solid reductant. Complete surrounding of pellet by solid reluctant was ensured. After attaining the reduction temperature each container was taken out at an interval of 15, 30, 45, 60, & 90 minutes for 800, 850 & 900^{0}C temperature and 5, 10, 15, 20, & 25 for 950^{0}C temperature. The containers were air cooled at room temperature and the loss of pellet’s weight was recorded. The degrees of reduction of pellets were calculated by using the following formula.

Degree of reduction = (weight loss in pellet/total oxygen content in the pellet) ×100

3.10 Determination of Percentage Swelling

Swelling is a volumetric expansion of the agglomerate during carbothermic reduction of iron oxide. Changes in crystal structure take place during the stepwise reduction of hematite through magnetite and wustite to metallic iron. Changes are accompanied with change in volume. Percentage swelling can be calculated as:

$$\% \, \boldsymbol{Swelling} = \frac{\boldsymbol{V_f - V_i}}{\boldsymbol{V_i}}$$

V_f – final volume of the reduced pellet

V_i – initial volume of the fired pellet.

Volumes of the pellets before and after the reduction were calculated by measuring the diameters of the pellets before and after the reduction.

CHAPTER-4

RESULTS&

DISCUSSIONS

4. RESULTS AND DISCUSSION

4.1 Characteristics of Selected Iron Ore and Coal

Sakaruddin Iron Ore chemical compositions and loss on ignition values were determined in Rourkela Steel Plant by X-Ray fluorescence technique and have been listed in Table 4.1.As outlined in Table 4.1, the selected iron ore is rich in Fe content(>60%). Al2O_3+ SiO_2 contents are less than 5% so, they are suitable for DRI production. Caking index of coal was found to be two which is suitable for to be used in sponge iron making, as is evident from Table 4.2 for the selected coal. The proximate analysis result reveals the fixed carbon content of coal is 40.11, ash content volatile matter is 27.26 and ash content is 32.63.The ash fusion temperature of the selected coal is high, which is in good agreement with the data for sponge iron making. The data for ash fusion temperatures have been taken from the work of kumar and patel [2].

4.2 Effects of Binder Content on the Crushing Strength and Porosity of the Pellets.

The Crushing Strength of fired pellets increases with increase in binder content. In the current project work the binder used is concentrated sugarcane juice. 2wt% binder content gave the minimum crushing strength and 6wt% binder content gave the maximum crushing strength. Porosity almost remains the same with increase in the binder content. This is quite clear from Tab 4.4 and figure 4.3 & 4.4.

4.3 Effect of Reduction Temperature on the Degree of Reduction and Extent of Swelling of Fired Iron Ore Pellets.

Degree of reduction increases with the increase in reduction temperature. An increase in the degree of reduction with reduction temperature appears to be due to more and more participation of gaseous reducing agents (CO and H_2) released from the devolatilization of coal and $C + O_2 \rightarrow 2CO$ reaction. The high rates of diffusion of gases through the metallic layer also contribute to this.

The extent of swelling first increases a little bit with the increase in reduction temperature after that it decreases with increase in reduction temperature. The initial increase may be due to high temperature, high degree of reduction and formation of fibrous structures. The decrease in the swelling after that with increase in the reduction temperature is more likely to be due to sintering of iron whiskers at higher temperature.

4.4 Effect of Reduction Time on Degree of Reduction and Extent of Swelling of Fired Iron Ore Pellets.

As shown in fig 4.5 & 4.8, the degree of reduction increases with increase in time at a particular reduction temperature. This increase in degree is due to the exposure of pellets with the reducing agents (C, CO, and H_2) for a longer period of time .The higher reduction rate in initial conditions may be attributed to the combined effect of less resistance offered to the flow of reducing gas into the pellet and significant contribution of volatile matter released initially.

As shown in fig 4.7 & 4.9 the extent of swelling increases with the increase in reduction time at a particular reduction temperature and particular firing temperature. This is believed to be due to fibrous growth of iron whiskers in the pellet matrix without any restriction in the growth, however the pellets fired at a higher temperature showed a decrease in the extent of swelling with increase in reduction time. This appears to be due to decrease in porosity due to better densification which restricts the fibrous growth of whiskers and prevents volume expansions.

4.5 Correlation between Degree of Reduction and Percentage Swelling

The variation of swelling (%) Vs Degree of Reduction (%) has been shown in figures (4.6 & 4.10) and in table 4.5. From the figures, it can be seen that the abnormal swelling (28-30%) was observed at around 90-95% reduction (FeO→Fe) at temperature 800^0C and 850^0C. It is expected to be due to the growth of iron whiskers. However Shrinkage in the reduced iron ore pellets was observed at 900^0C and 950^0C. This is expected to be due to the sintering of iron at high temperatures.

4.6 XRD Analysis of Reduced Iron Ore Pellets.

The XRD patterns of sakaruddin iron ore pellets reduced at temperatures of 800, 850, 900 and 950^0C for a time period of 0, 60, 15, 30 minutes respectively by Basundhara non-coking coal, which is shown in figure 4.11 and the major & minor phases at various temperatures is shown in table 4.3. It is quite clear from the fig.4.11 that only one peak of FeO present in the XRD patterns of reduced iron ore pellet. In general, the amount of Feo content in the reduced pellet decreased with increase of reduction temperature.

4.7 Scanning Electron Micrographic Study of the Reduced Pellets.

As shown in fig 4.17- 4.18 cracks are developed in the iron ore pellets due to swelling, the cracks formed in the reduced pellets are more pronounced at 850^{o}C as compared to Cracks at 800^{0}C, 900^{o}C and 950^{o}C because of densification due to reduction at high temperatures.

Densification of the iron ore particles has occurred. Densification is much more in case of reduction at 950^{o}C as compared to the other lower reduction temperatures.

The excessive swelling of the iron ore pellets at lower temperatures is due to fibrous growth of iron whiskers and formation of more cracks in the pellet matrix, which is also visible in the SEM photographs.

TABLES

Table 4.1

Chemical Composition and Loss on Ignition of Iron ore

Iron Ore Source	**Chemical Composition (weight percent on dry basis)**						
	Fe (Total)	Fe_2O_3	Al_2O_3	SiO_2	TiO_2	MnO	LIO*
Sakaruddin Mine	65.50	93.01	2.38	1.36	0.15	0.02	3.08

LIO*=LOSS ON IGNITION

Table 4.2

Proximate Analysis, Reactivity, Caking Indices, Gross Calorific Value and Ash Fusion Temperature of Non-coking Coal

Non-coking Coal	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)	Sulphur content (%)	Caking -Index	Ash Fusion Temperature (^{0}C)			
						IDT	**ST**	**HT**	**FT**
Basundhara Mine	27.26	32.63	40.11	0.48	2.0	1253	1428	1524	1600

Table 4.3

XRD Analysis of Reduced Sakaruddin Iron Ore Pellets

Temperature (0C)	Time (minutes)	Major Phase	Minor Phase
800	**60**	**Fe**	**FeO**
850	**45**	**Fe**	**FeO**
900	**15**	**Fe**	**FeO**
950	**30**	**Fe**	**FeO**

Table 4.4

Results of Physical Properties of Sakaruddin Fired Iron Ore Pellets

Binder	Binder (%)	Firing Conditions		Drop No		Crushing strength Kg/ Pellet	Porosity (%)
		Firing Temp(°C)	Firing Time(hr.)	Oven dried	Fired		
Concentrated Sugarcane juice	2	1100	01	01	08	55	22.5
	4	1100	01	02	421	205	22.58
	6	1100	01	04	622	240	21.4

Table 4.5

Results of Degree of Reduction and Swelling Values of Fired Sakaruddin Haematite Iron ore pellets Reduced in Basundhara non-coking coal.

Binder	Binder (%)	Pellet composition: –100# (95%), –16+25# (5%)					
		Firing Conditions		Reduction Conditions		Degree of Reduction (%)	Swelling (%)
		Firing Temp(°C)	Firing Time(hr.)	Reduction Temp(°C)	Reduction Time(Min)		
Concentrated Sugarcane juice	2	1100	1	800	15	53.81	5.26
					30	71.80	10.9
					45	83.50	6.80
					60	82.52	3.17
					90	89.05	4.05
				850	15	65.70	3.40
					30	74.60	6.20
					45	78.88	4.26
					60	86.72	2.02
					90	91.52	2.09
				900	15	72.50	5.32
					30	73.60	6.59

					45	**79.40**	**2.92**
					60	**82.26**	**6.54**
					90	**88.99**	**7.88**
				950	**5**	**72.60**	**6.12**
					10	**75.30**	**6.07**
					15	**97.08**	**3.30**
					20	**99.88**	**4.40**
					25	**98.20**	**2.46**
Concentrated Sugarcane Juice	**4**	**1100**		**800**	**15**	**50.81**	**5.16**
					30	**74.80**	**9.9**
					45	**85.50**	**5.80**
					60	**84.52**	**4.17**
					90	**88.05**	**4.15**
				850	**15**	**66.70**	**3.50**
					30	**77.60**	**6.80**
					45	**75.88**	**5.26**
					60	**88.72**	**3.02**
					90	**89.52**	**2.19**
				900	**15**	**73.50**	**5.92**
					30	**74.60**	**6.99**
					45	**82.40**	**3.92**
					60	**79.26**	**6.94**
					90	**90.99**	**7.18**
				950	**5**	**70.60**	**7.12**
					10	**76.30**	**6.97**
					15	**98.08**	**3.90**
					20	**99.88**	**4.90**
					25	**97.20**	**2.46**

Figures

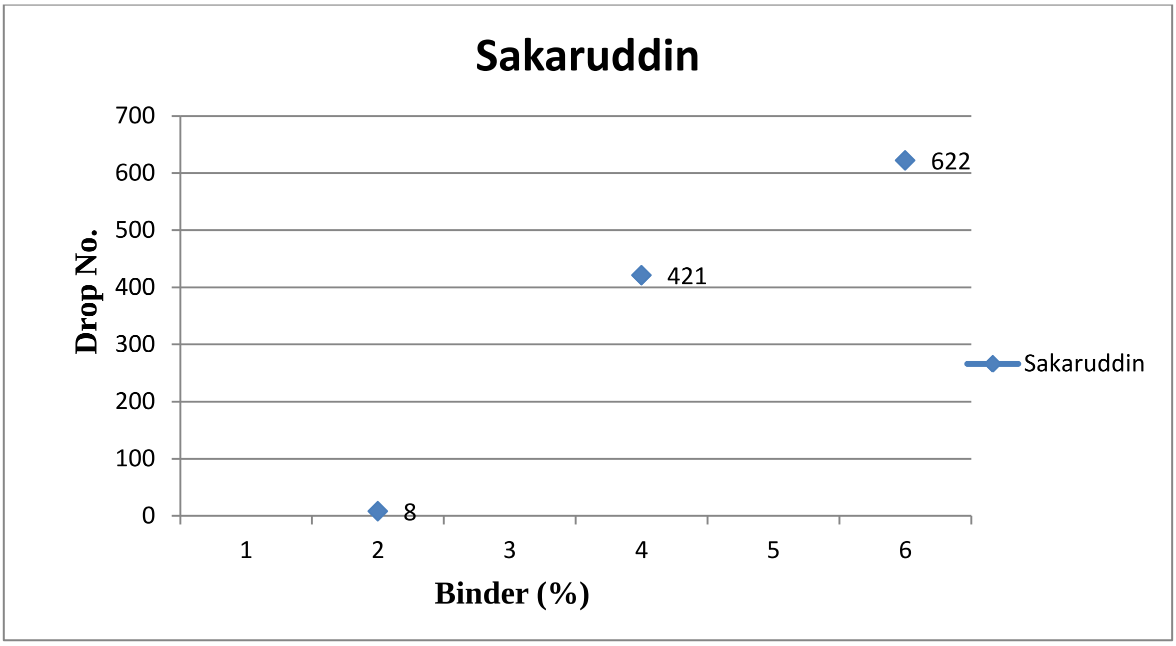


Figure 4.1: Effect of binder content on the Drop No. of fired Sakaruddin haematite iron ore pellets [2% binder, {-100#(95%), -16+25(5%)}].

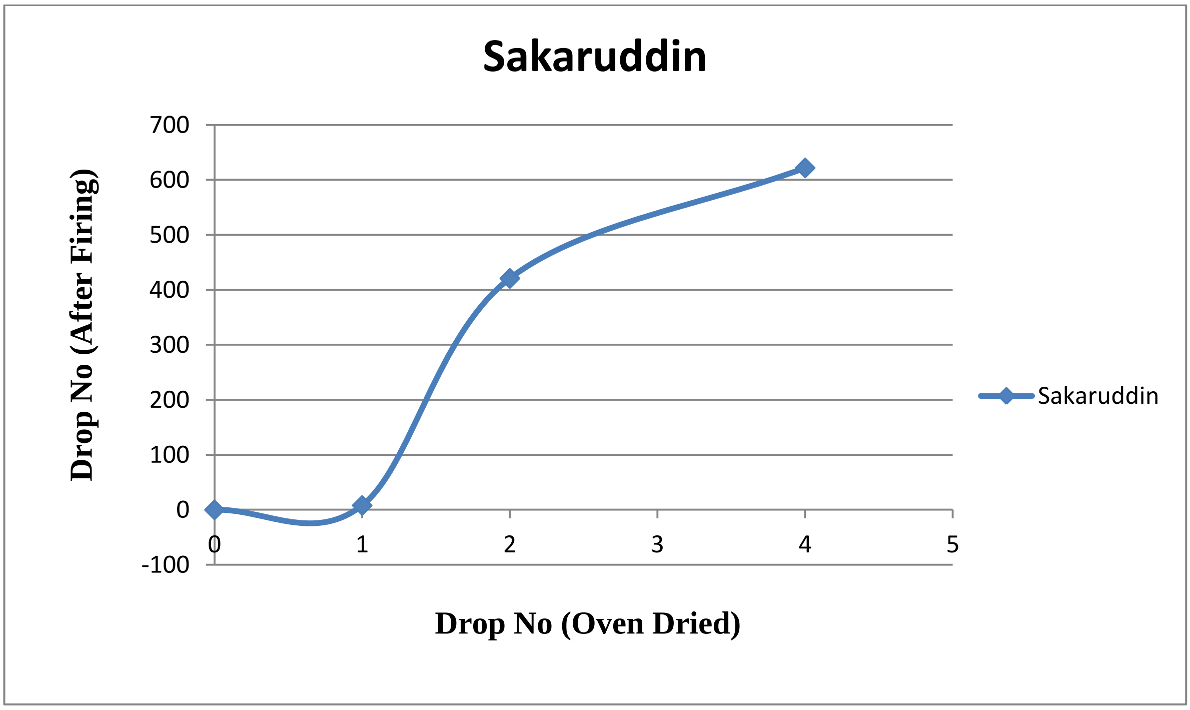


Figure 4.2: Effect of Drop No. of oven dried content on the Drop No. of fired Sakaruddin haematite iron ore pellets [2% binder, {-100#(95%), -16+25(5%)}].

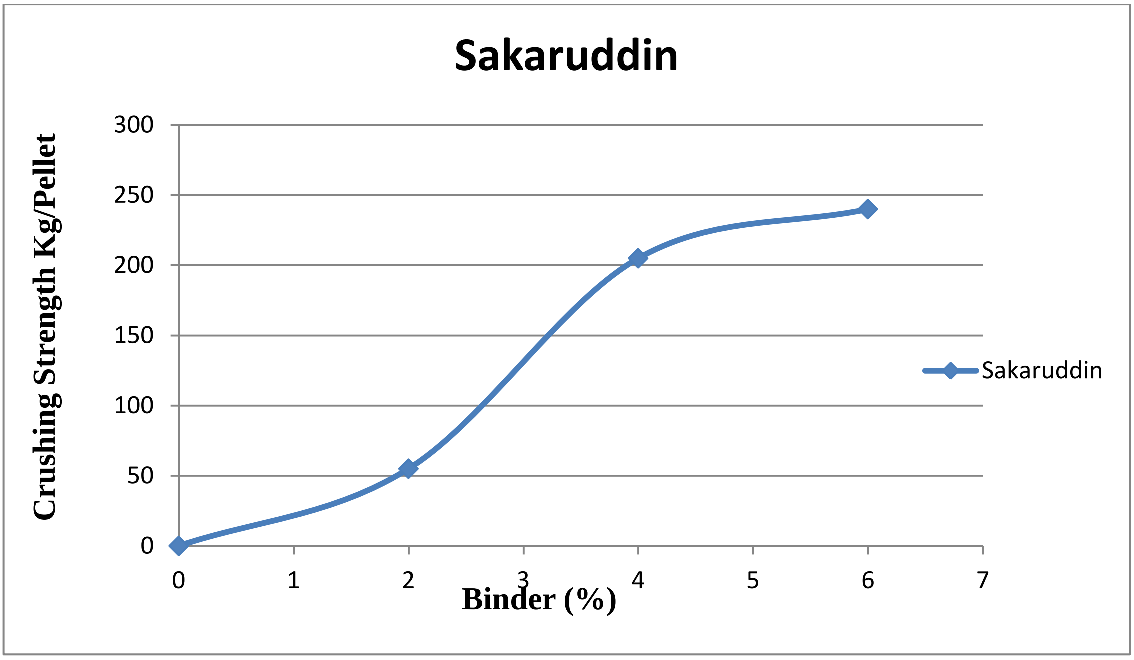


Figure 4.3: Effect of binder content on the Crushing Strength of fired Sakaruddin haematite iron ore pellets [2% binder, {-100#(95%), -16+25(5%)}].

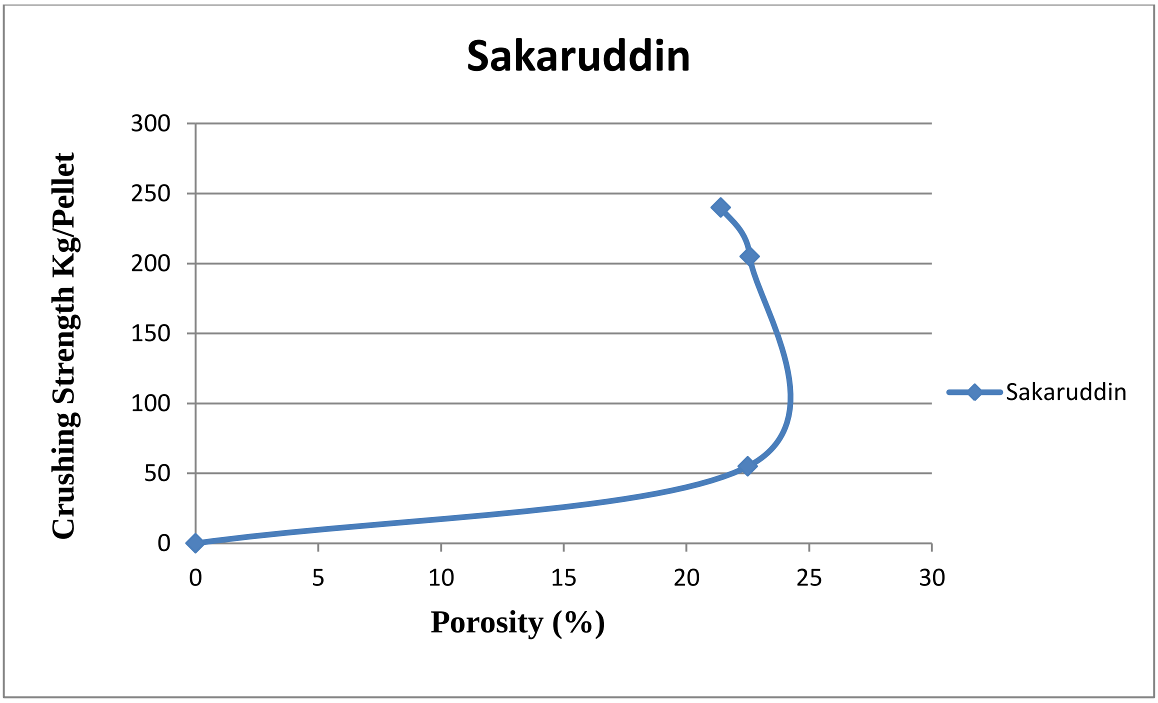


Figure 4.4: Effect of Porosity (%) on the Crushing Strength of fired Sakaruddin haematite iron ore pellets [2% binder, {-100#(95%), -16+25(5%)}].

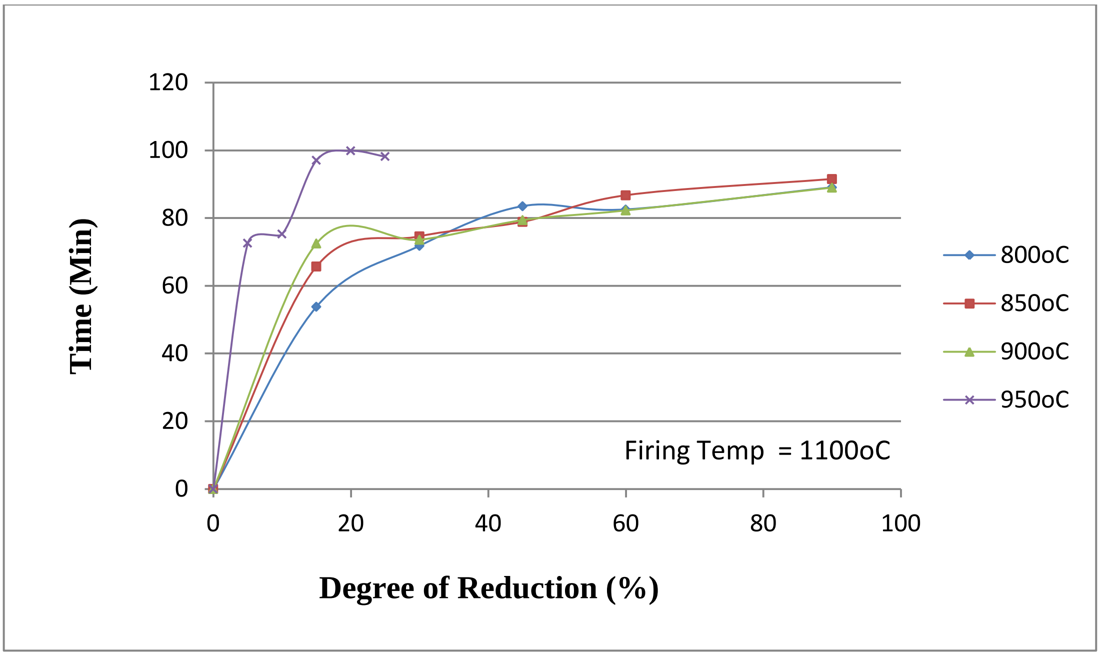


Figure 4.5: Effects of Time on Degree of Reduction of Sakaruddin iron ore pellets [2% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

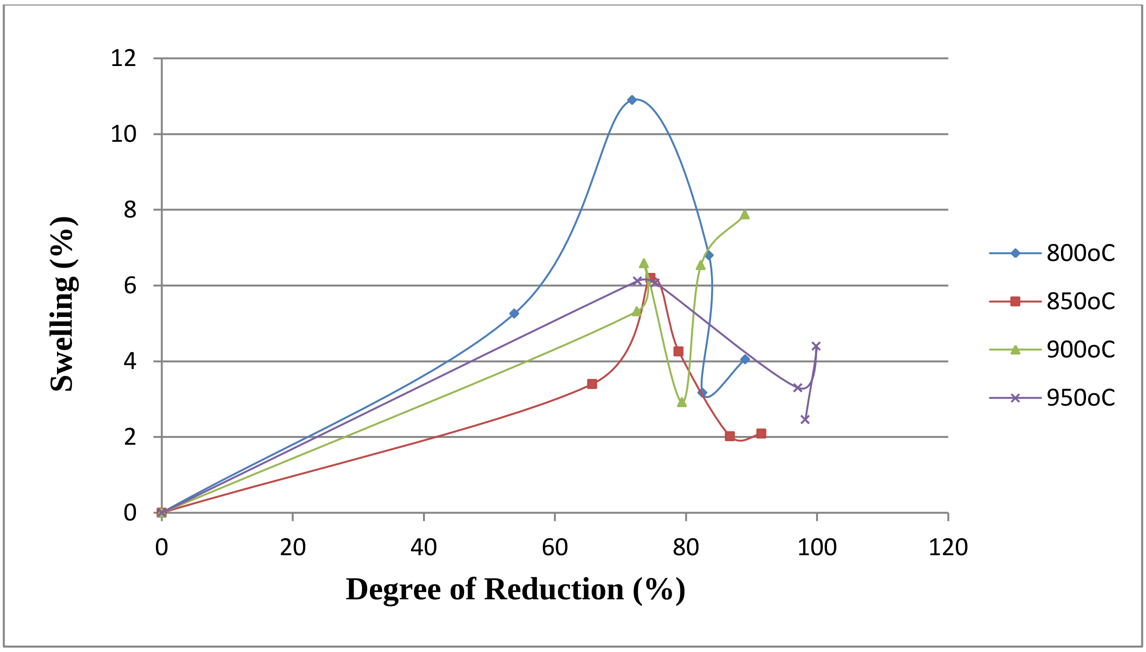


Figure 4.6: Correlation between Swelling (%) and Degree of Reduction of Sakaruddin iron ore pellets [2% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

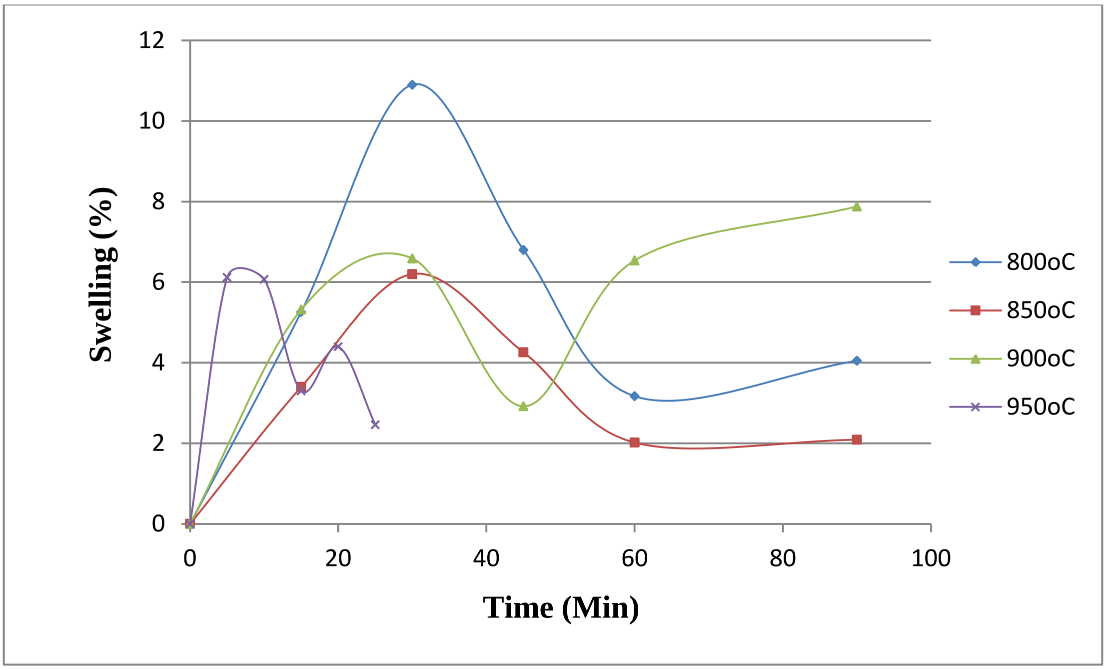


Figure 4.7: Effects of Reduction Time (min) on Swelling (%) of Sakaruddin iron ore pellets [2% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

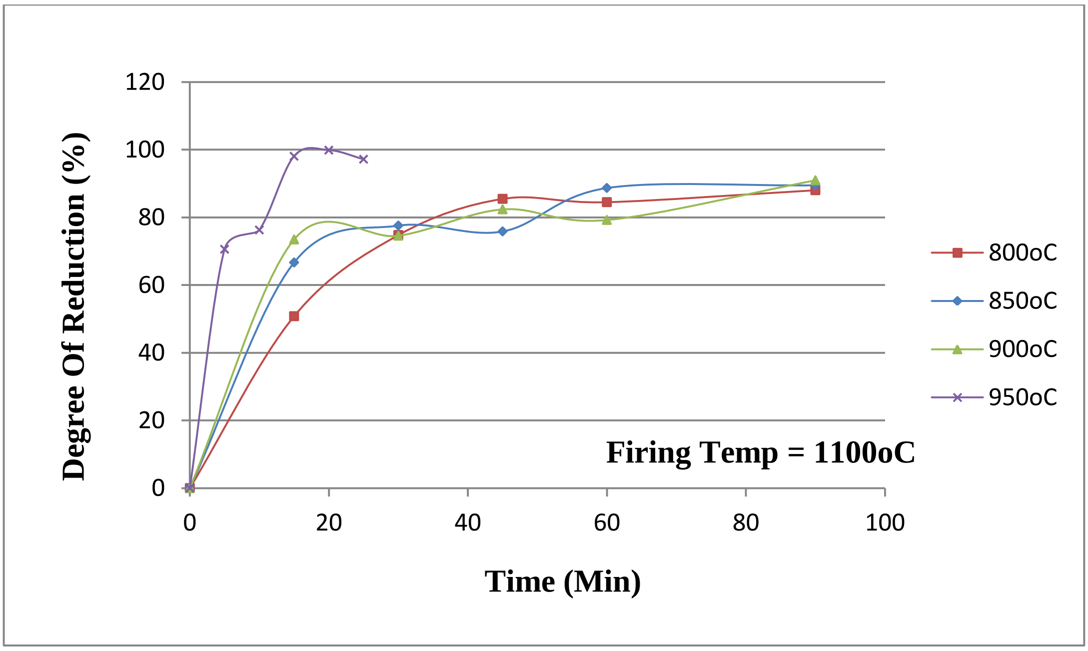


Figure 4.8: Effects of Time on Degree of Reduction of Sakaruddin iron ore pellets [4% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

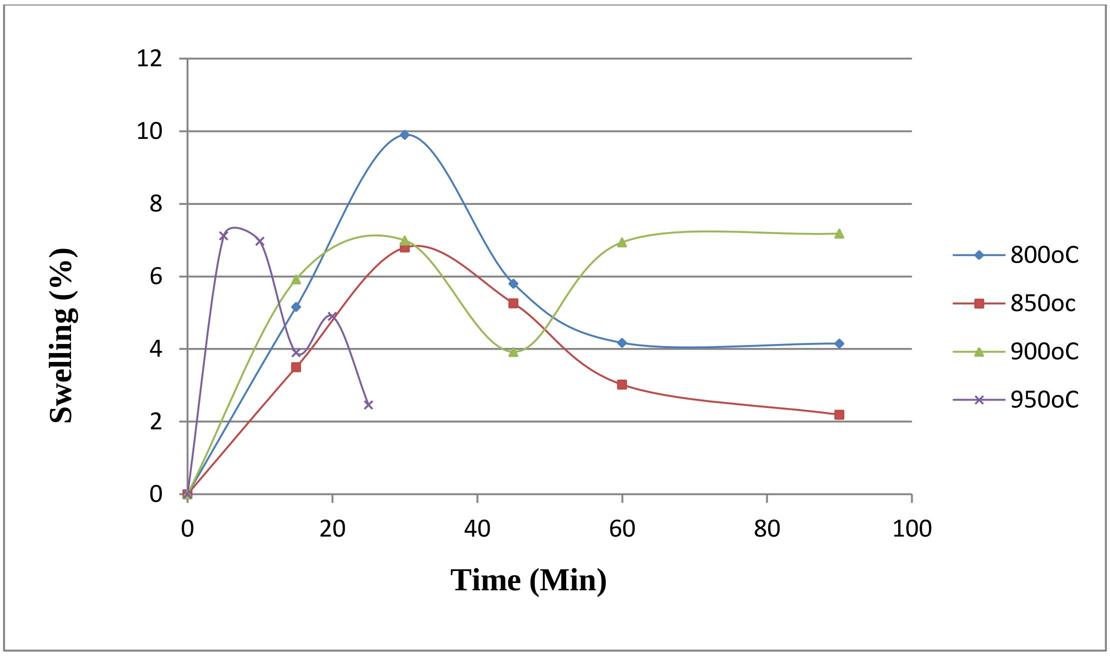


Figure 4.9: Effects of Reduction Time (min) on Swelling (%) of Sakaruddin iron ore pellets [4% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

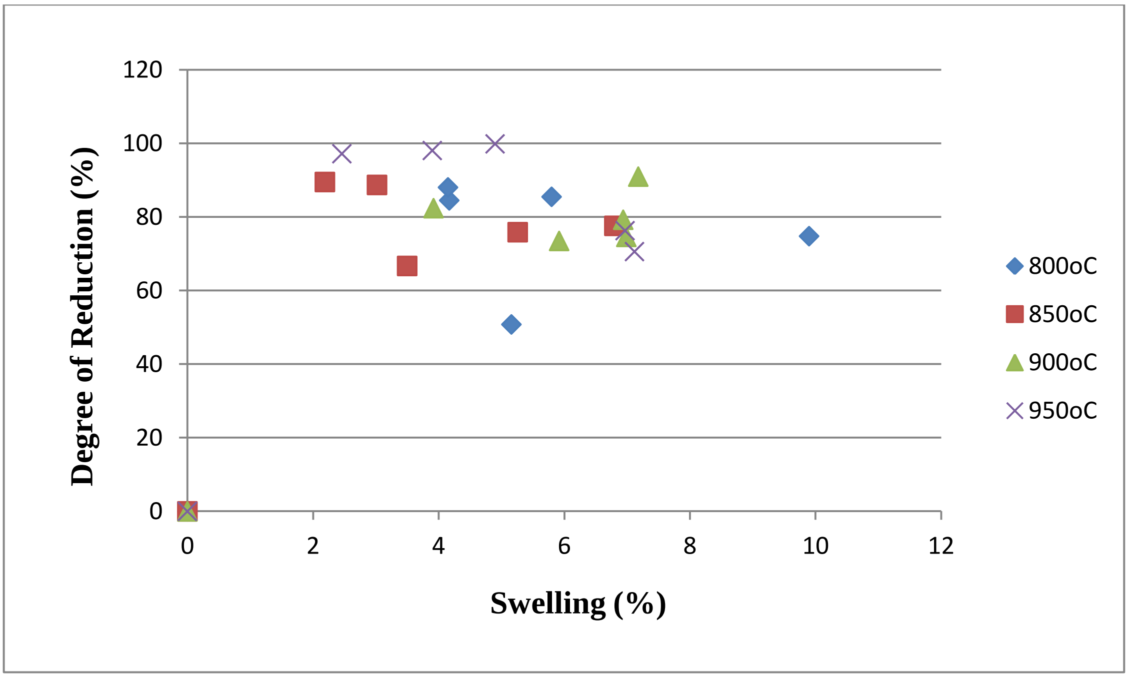


Figure 4.10: Relationship between Swelling (%) and Degree of Reduction of Sakaruddin iron ore pellets [4% binder, firing temperature-1100°C,{-100#(95%), -16+25(5%) }].

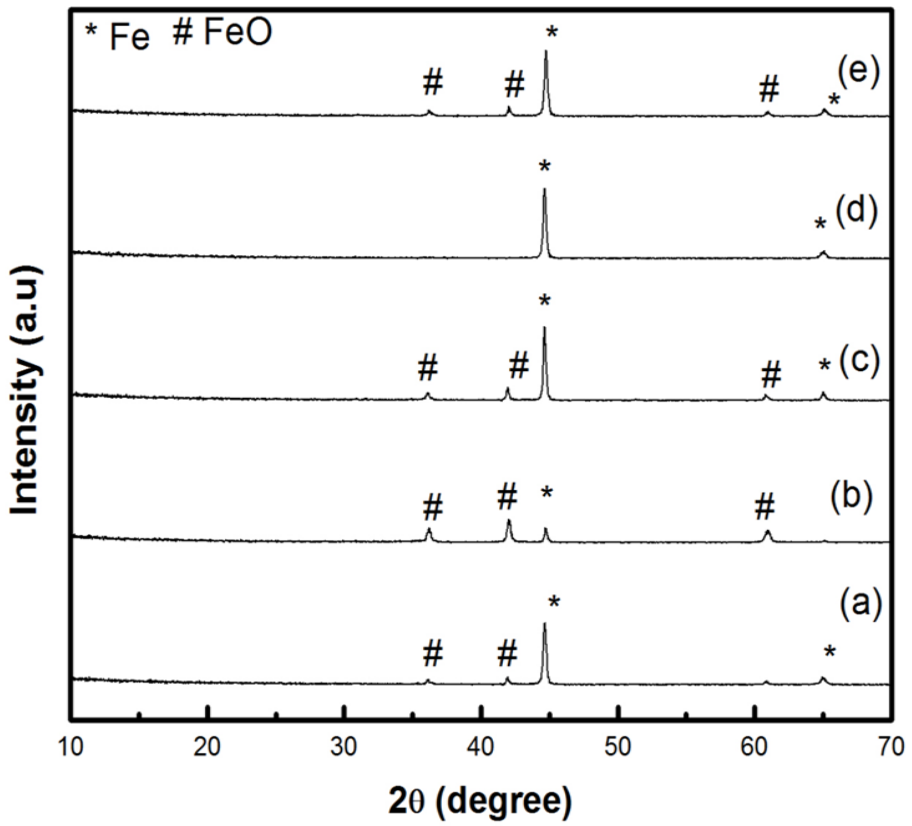
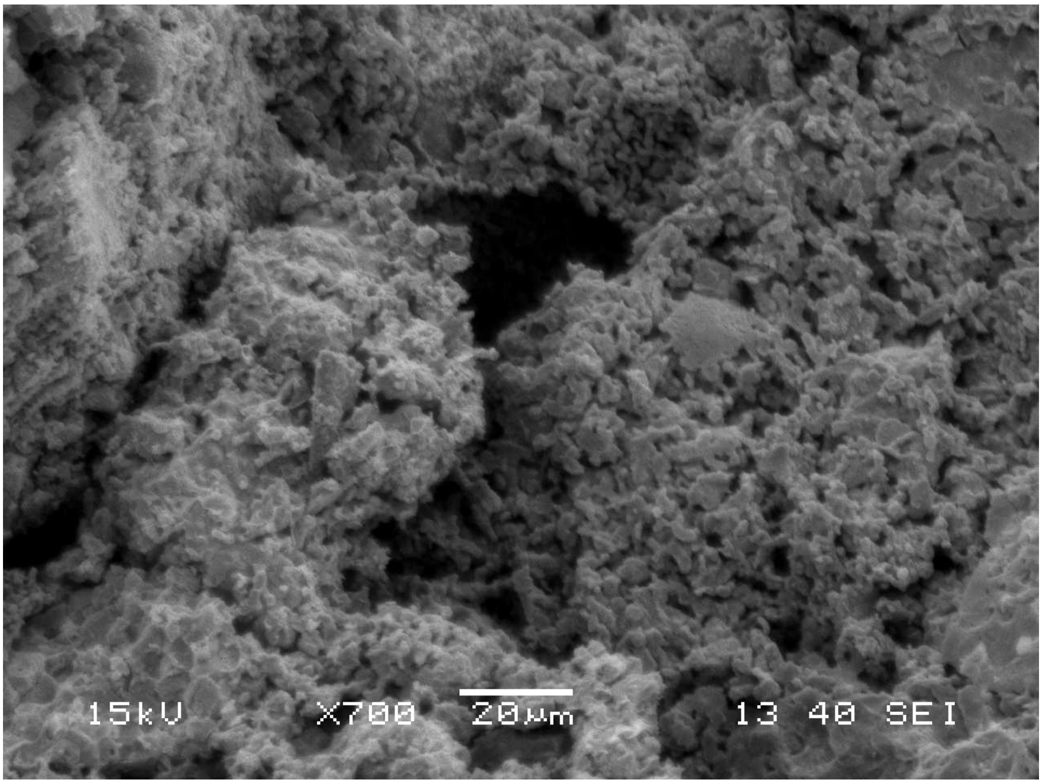


Fig-4.11 XRD Patterns of sakaruddin iron ore pellets reduced at different temperature in Bashundhara non-coking coal (a)Temp.-950°C, Time- 30 min (b)Temp. -900°C, Time- 15 min (c) Temp.-850°C, Time- 45 min (d) Temp.-850°C, Time- 60 min (e) Temp.-800°C, Time- 90min.



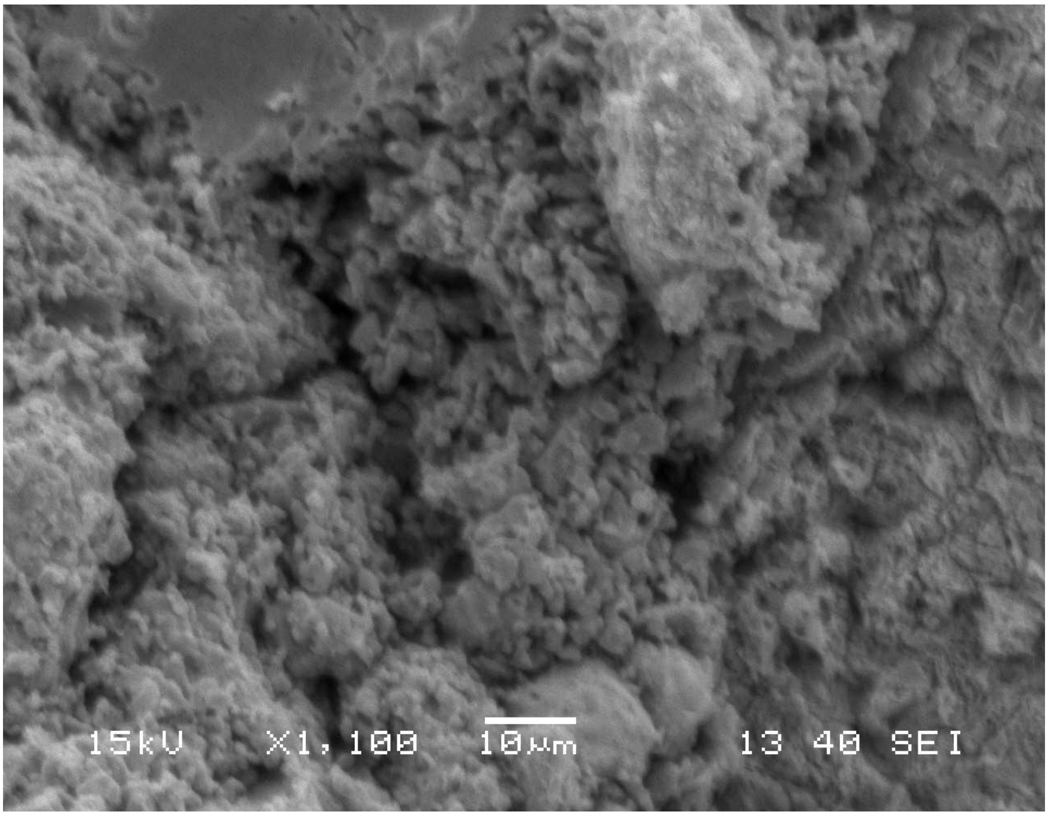
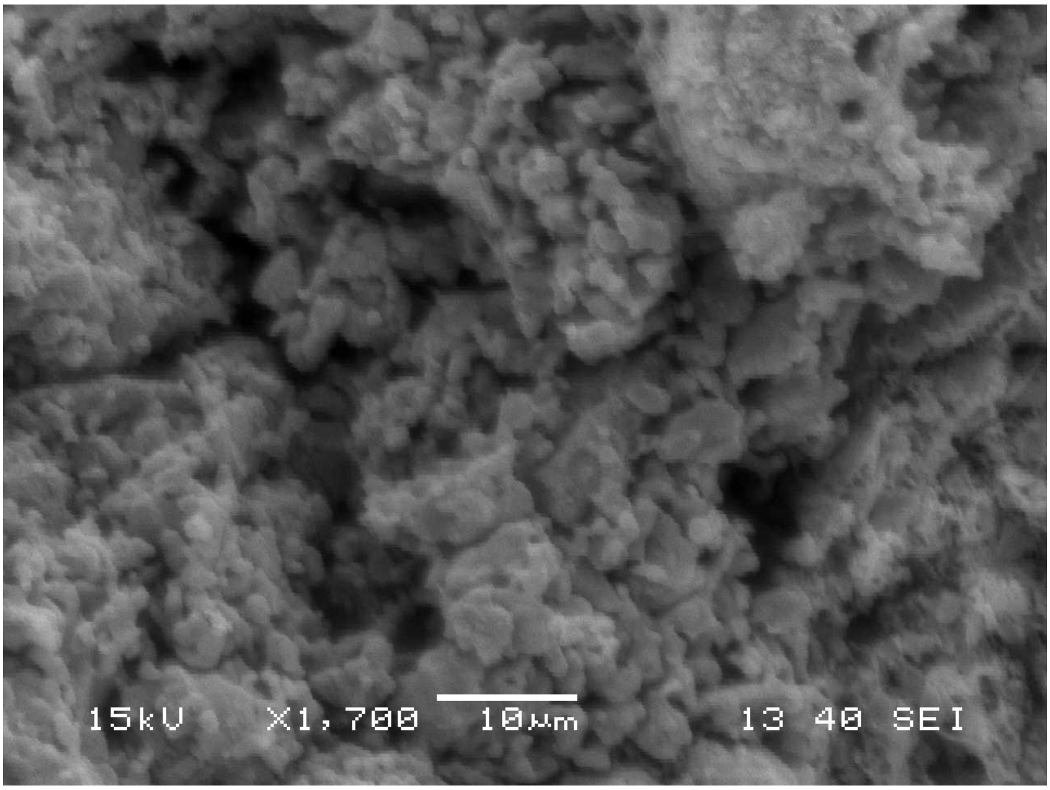


Fig 4.12 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-950°C, 2% binder, reduction time-30 min,{-100#(95%),-16+25#(5%)}].



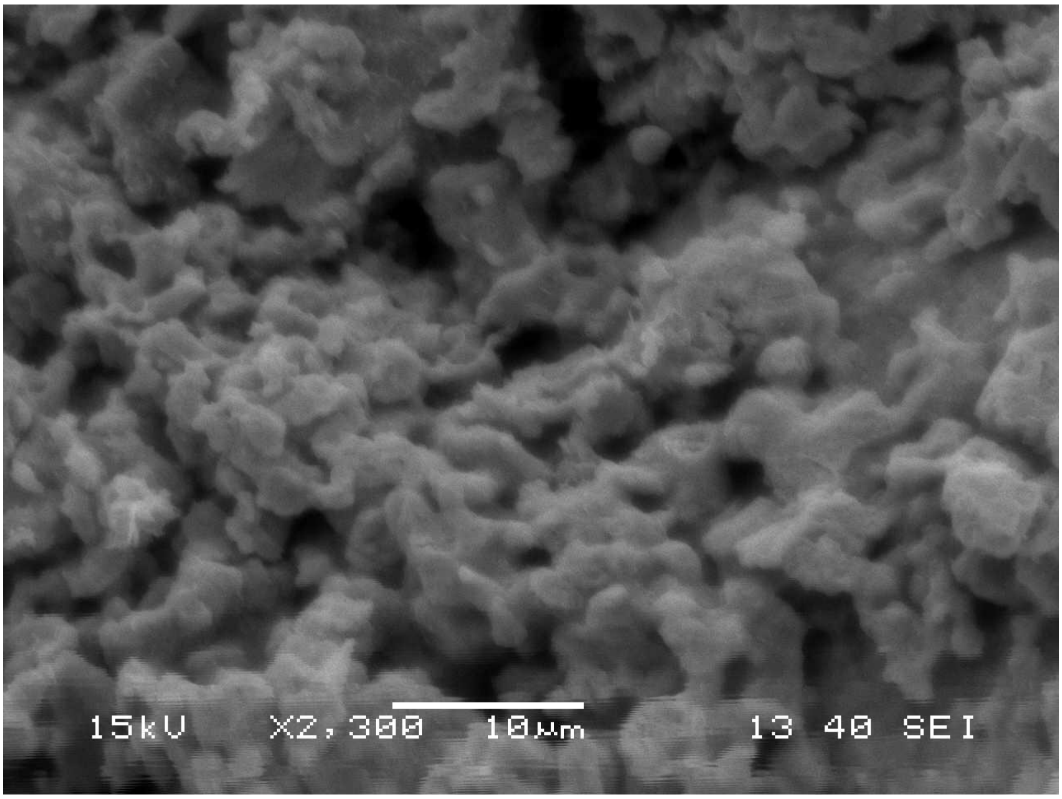
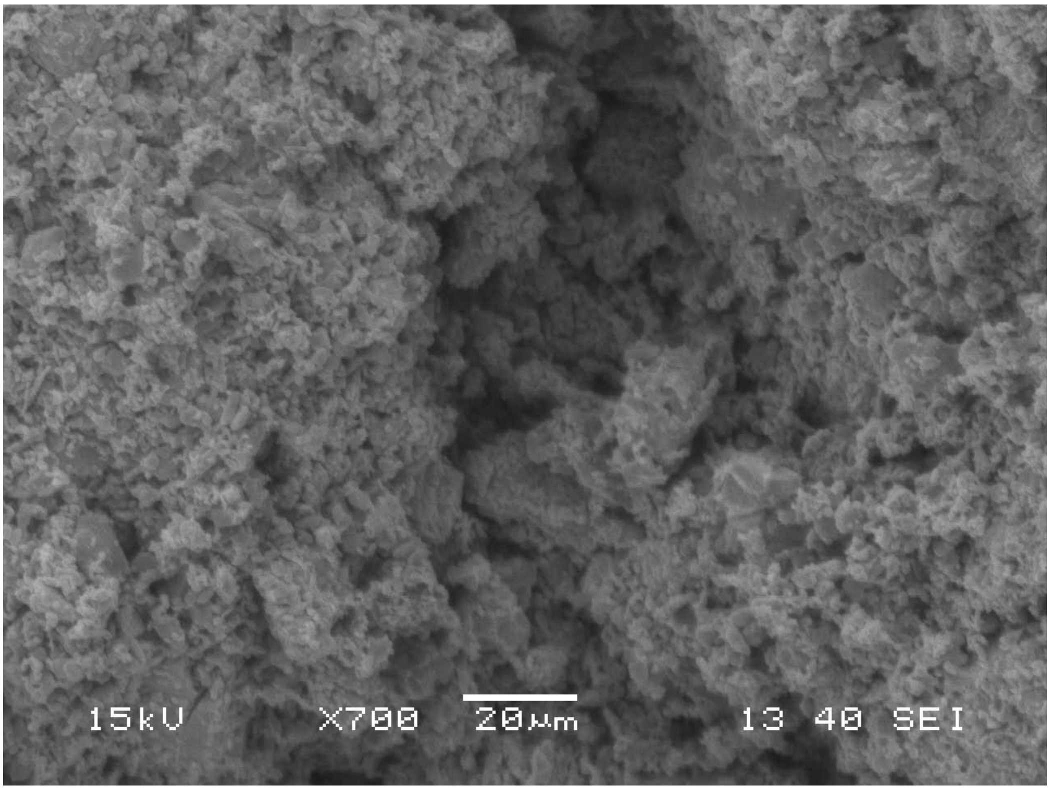


Fig 4.13 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-950°C, 2% binder, reduction time-30 min,{-100#(95%),-16+25#(5%)}].



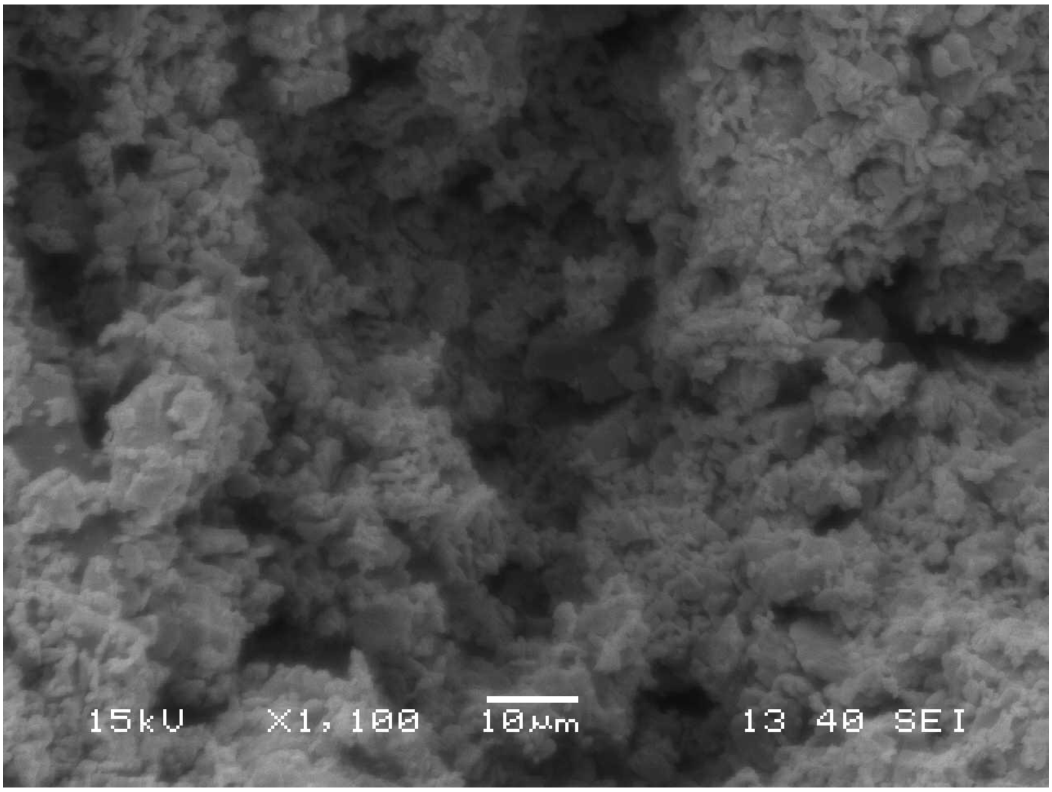
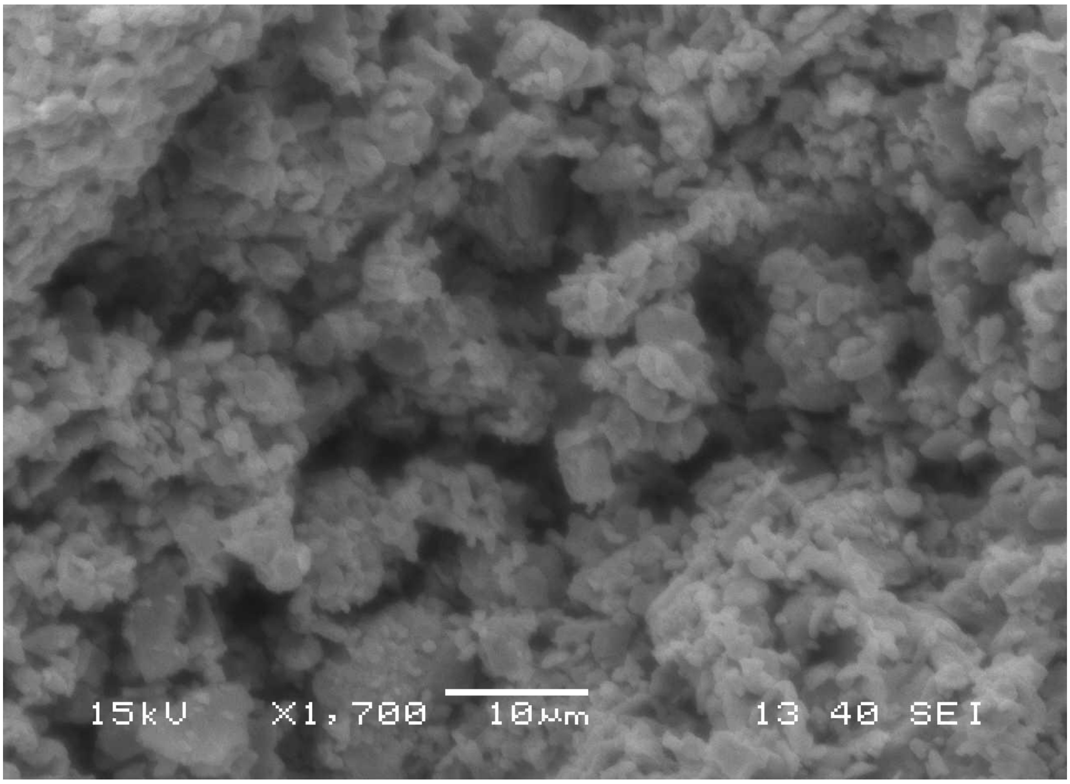


Fig 4.14 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-900°C, 2% binder, reduction time-15 min,{-100#(95%),-16+25#(5%)}].



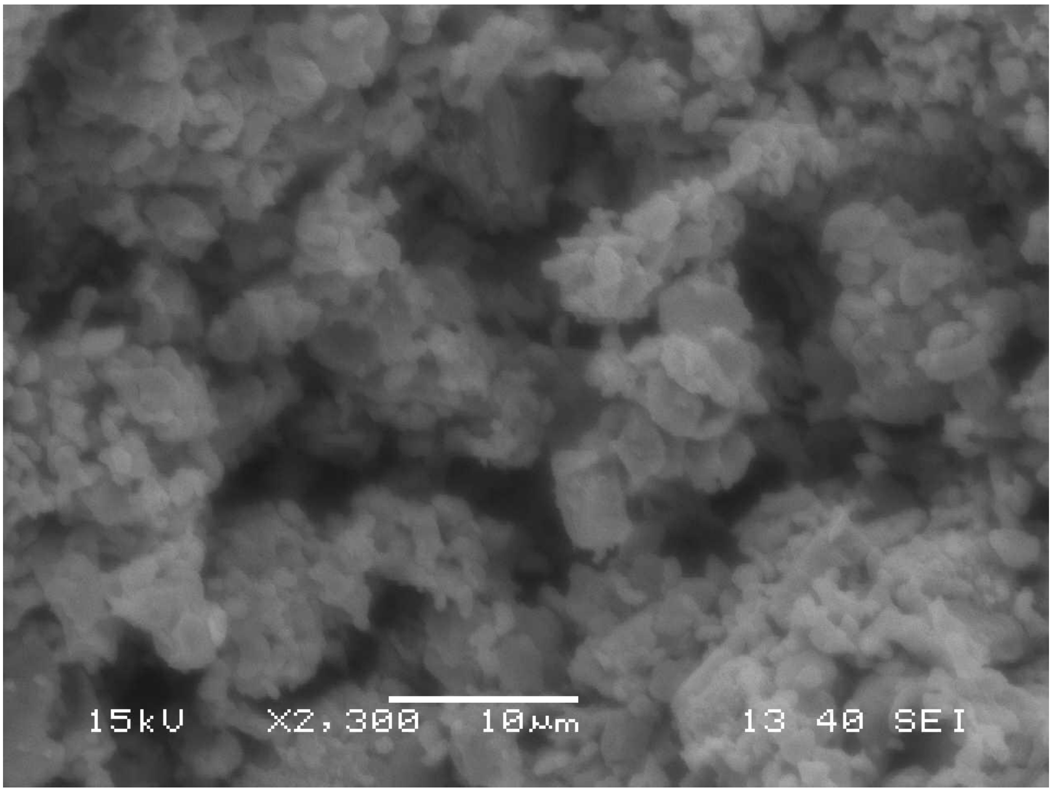


Fig 4.15 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-900°C, 2% binder, reduction time-15 min,{-100#(95%),-16+25#(5%)}].

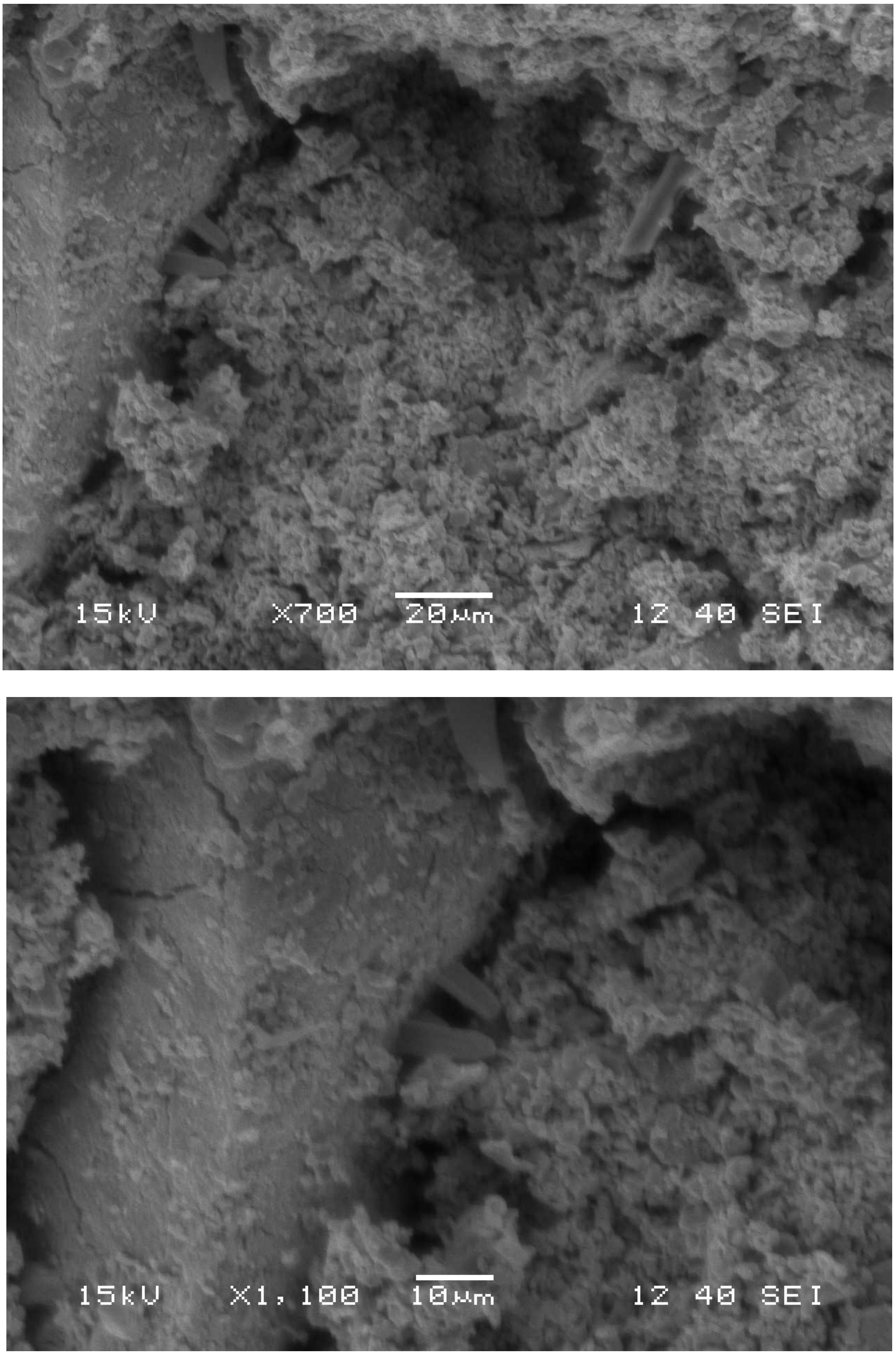
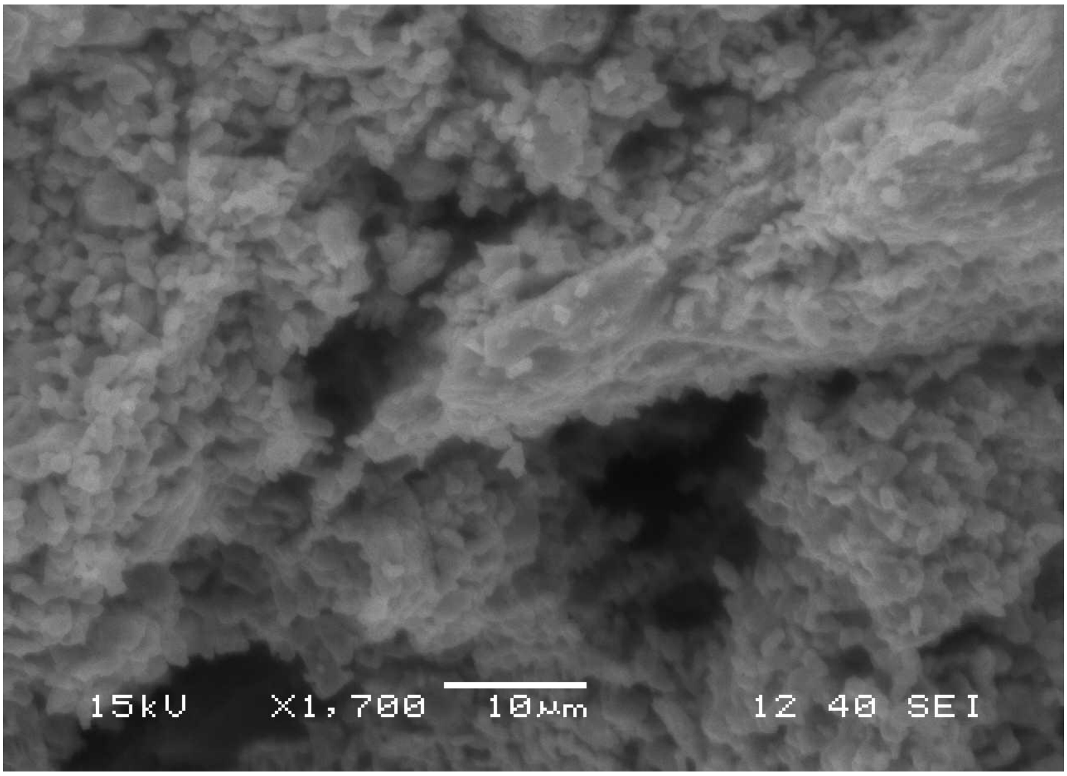


Fig 4.16 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-850°C, 2% binder, reduction time-45 min,{-100#(95%),-16+25#(5%)}].



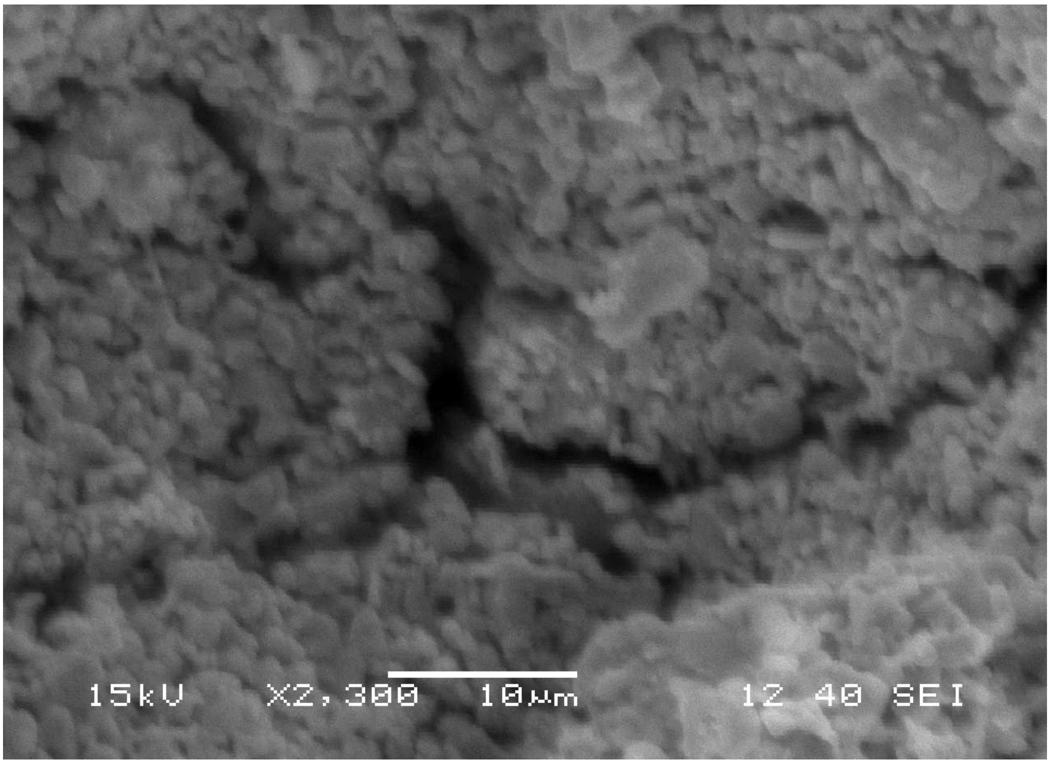
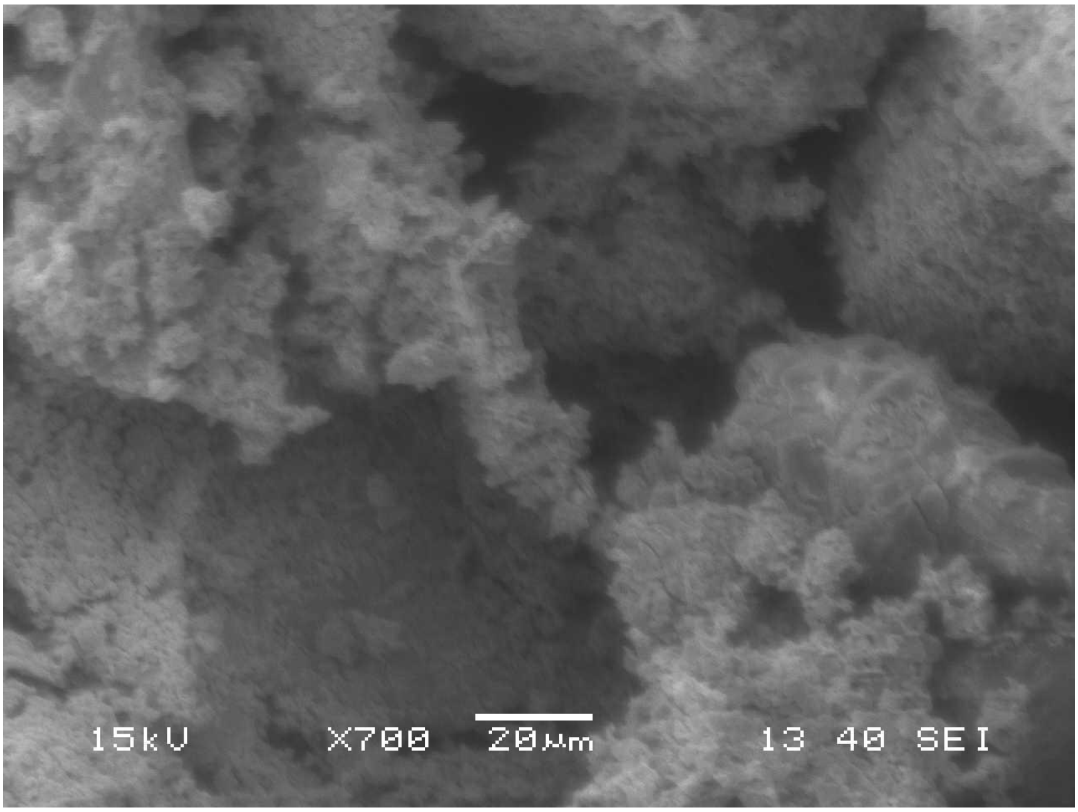


Fig 4.17 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-850°C, 2% binder, reduction time-45 min,{-100#(95%),-16+25#(5%)}].



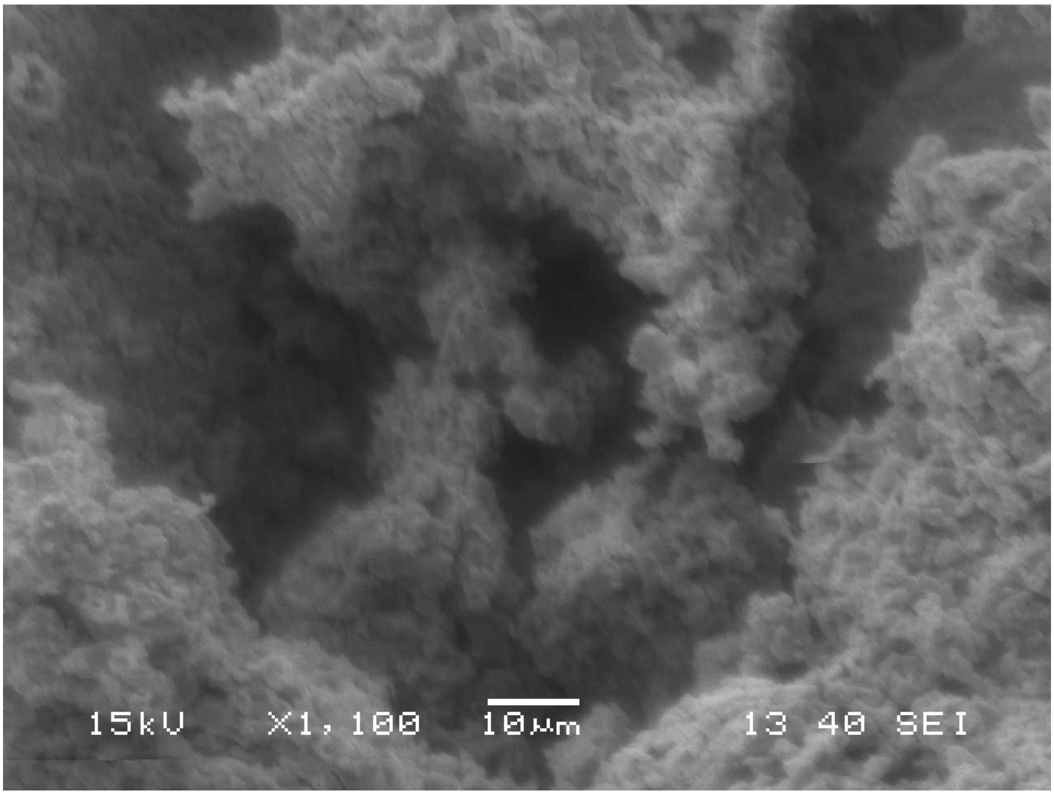


Fig 4.18 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-800°C, 2% binder, reduction time-90 min,{-100#(95%),-16+25#(5%)}].

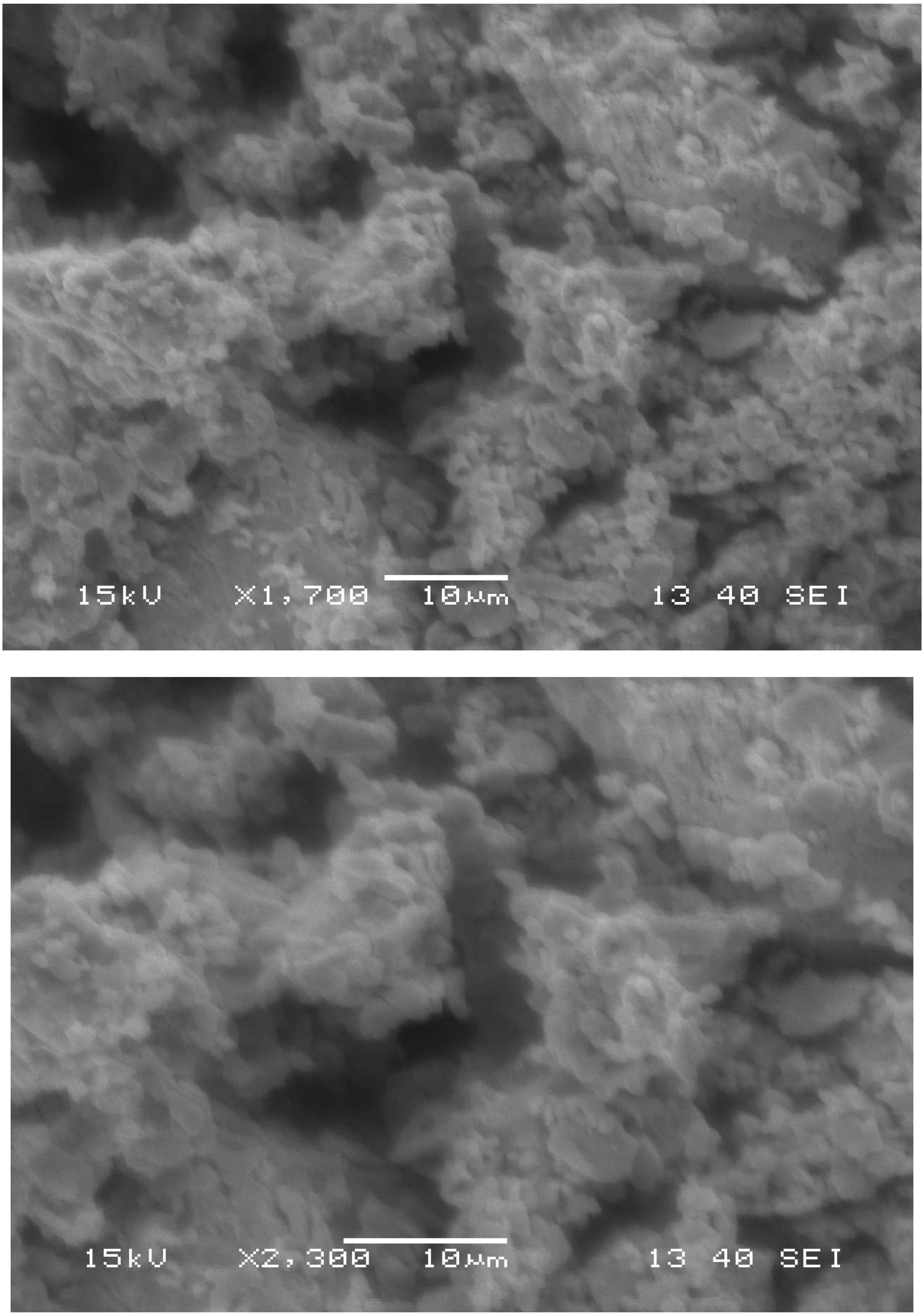


Fig 4.19 SEM photograph of reduced Sakaruddin haematite iron ore pellet fired at 1100°C for 1 hour [reduction temperature-800°C, 2% binder, reduction time-90 min,{-100#(95%),-16+25#(5%)}].

CHAPTER-5

CONCLUSION

CONCLUSIONS

From the results of the present project work, the following conclusions may be drawn.

1) The Crushing Strength of fired hematite iron ore pellets vary with the variation of binder content and minimum strength found for 2% (weight percentage) and maximum strength found for 6% (weight percentage) of binder content.

2) The crushing strength of pure -100# fired iron ore pellets is high compared to the pellets made with blending of different size iron ore fines.

3) Degree of reduction increases with increase in reduction temperature due to more diffusion of reducing gases and carbon in the pellet matrix.

4) The extent of swelling first increases and then decreases with increase in reduction temperature, their abnormal swelling in temperature 800^0C & 850^0C whereas shrinkage was found in temperature 900^0C & 950^0C.

5) The degree of reduction increases with increase in time at a particular reduction temperature.

6) This is of greatest advantage in the use of these iron ore pellets in rotary kiln for sponge iron production, which can lead to a saving of enormous amount of energy.

7) With increase in binder content, drop numbers increase.

8) XRD results confirmed that Feo content in the reduced pellet, In general, decreased with increase in reduction temperature.

9) SEM photographs revealed more densification in the pellets reduced at higher temperatures.

CHAPTER-6

FUTURE WORK

FUTURE SCOPE OF THE PROJECT:

The works carried out in this area may be extended in the future by other investigators. The suggested future work is as follows-

1. Detailed studies on reduction and swelling behaviour of fired iron ore pellets may be carried out with other non-coking coals and iron ores of Orissa, Jharkhand, Chhattisgarh and nearby areas.

2. This work may be extended for study on reduction and swelling behaviour of iron-ore lumps.

3. Similar studies may be carried out with other binders and iron ore fines.

CHAPTER-7

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